**REPORT NO. MA 19-02** | Wenche Emblem Larssen, Mats Carlehög, Trygg Barnung, John-Erik Haugen, Kari Fjørtoft, Wenche Uksnøy, Charlotte Jacobsen, Erminio Monteleone and Margrethe Hersleth

# CLASSIFICATION OF FISH OIL THROUGH STUDY OF THEIR SENSORY AND CHEMICAL PROPERTIES

MØREFORSKING

UNIVERSITÀ DEGLI STUDI FIRENZE

Nofima

DTU

| | |
|---|---|
| **TITLE** | Classification of fish oil through study of their sensory and chemical properties |
| **AUTHORS** | Wenche Emblem Larssen, Mats Carlehög, Trygg Barnung, John-Erik Haugen, Kari Fjørtoft, Wenche Uksnøy, Charlotte Jacobsen, Erminio Monteleone and Margrethe Hersleth |
| **PROJECT LEADER** | Wenche Emblem Larssen |
| **REPORT NO.** | MA 19-02 |
| **PAGES** | 62 |
| **PROJECT NO.** | 54904 |
| **PROJECT TITLE** | QOMEGA3-Sensorisk bransjestandard for fiskeoljer |
| **CONTRACTING** | The Norwegian Seafood Research Fund (FHF) |
| **RESPONSIBLE PUBLISHER** | Møreforsking Ålesund AS |
| **ISSN** | 0804-5380 |
| **DISTRIBUTION** | Open |
| **KEYWORDS** | Oxidation, sensory characteristics, standardization, classification |



## SUMMARY

This study has shown that the sensory characteristics of fish oils give an accurate and representative description of the quality of the oils and that a common sensory standard may be a valuable tool in the industries' quality control and marketing. A classification system of the fish oils is defined, providing the industry with a simple and convenient tool in the industry's communication with their customers. Samples with low primary and secondary oxidation were associated with sensory attributes like *sourness* and *grass*, while oils with higher oxidation values were associated with sensory attributes like *rancid*, *fermented* and *process*. The sensory characteristic *fish* is defined as the fresh odour and flavour of fish. This attribute is allowed in all classifications, but at a low intensity. In a further study it may be beneficial to produce synthetic reference oils to train the sensory industries panels.

## SAMMENDRAG

Gjennom prosjektet har en sett at de sensoriske egenskapene til fiskeoljer gir en nøyaktig og representativ beskrivelse av oljens kvalitet, og at en felles sensorisk standard kan være et verdifullt verktøy i bransjens kvalitetskontroll og markedsføring. Et klassifiseringssystem for fiskeoljene er definert. Dette gir industrien et enkelt og praktisk verktøy for kommunikasjon ut mot kundeleddet. Prøver med lav primær og sekundær oksidasjon var forbundet med de sensoriske egenskapene syrlig og gress. Disse egenskapene er sammen med *nøtt og frø* og *smør* godkjente lukter og smaker i fiskeoljene. Oljer med høyere oksidasjonsverdier var assosiert med de sensoriske egenskapene *harsk, fermentert* og *prosess* og disse kan ikke være tilstede i en gull olje (høyeste klassifisering) og bare i svak eller moderat styrke i de lavere klassifiseringene. Lukt og smak av fersk fisk er tillatt i alle klassifikasjoner, men med lav intensitet. I en videre studie kan det være fordelaktig å utvikle syntetiske referanseoljer med spesifikke lukt- og smaksegenskaper. Disse kan benyttes under trening og kalibrering av de sensoriske industripanelene.

## PREFACE

The research project 'QOMEGA3 – Sensory industry standard for marine oils' is funded by The Norwegian Seafood Research Fund (FHF) and is a cooperation between Møreforsking Ålesund AS (MFAA), Nofima AS, Technical University of Denmark (DTU) and University of Florence (UniFI). A company cluster consisting of 9 omega-3 companies has been active in the project process. Thanks to Anja Helen Haugom at Marine Ingredient AS, Marte Grimstad at Berg Lipid Tech AS, Margrete Mosen and Siri Søvik at Orkla Health AS, Helga Midtkandal and Iren Stoknes at Epax Norway AS, Hanne Solvang Felberg and Bente Foss at GC Rieber Oils AS, Karin Fareth and Stig Janson at Nordic Pharma AS, Åse Christine Mikalsen at Calanus AS and Ingjerd Lystad at Pharma Marine AS. In addition, Blue Legasea with Wenche Uksnøy and Global Organization for EPA and DHA (GOED) with Gerhard Bannenberg have been valuable participants.

Thanks to all!

Ålesund, January 2019

Wenche Emblem Larssen

## INDEX

# 1. BACKGROUND

The Norwegian omega-3 industry is exposed to increasing competition from manufacturers from regions such as Asia and South America and needs differentiation tools that can highlight the unique quality of Norwegian-produced omega-3 oils.

At present, there are no fixed standards or requirements connected to sensory quality, and the industry wishes to develop a standard to offer its customers a taste guarantee for omega-3 products. Such a standard will provide internal value to companies in their quality control work and will be an important sales tool. A sensory industry standard will also provide competitive advantages to the Norwegian marine oil industry on the global scene. Marine oils oxidize (become rancid) easily, and lipid oxidation is one of the main causes of quality deterioration (Olsen 2005, Larssen et al. 2018). The oxidation process contributes to a change in smell and taste (Ruyter et al. 2010), and traditional fish oil and omega-3 products are therefore often associated with a rancid taste. For many years, the industry has made an effort to change this with the aim to provide its customers with a taste guarantee according to the industry's chemical quality requirements. A taste guarantee can help to build the reputation of the omega-3 industry and ensure that customers have confidence in the products.

A rancid smell and taste can be discovered sooner using sensory analyses than through the identification of traditional oxidation products such as peroxide and anisidine (Jacobsen 1999, Olsen 2005, Aas et al. 2016). Good procedures and tools for conducting sensory analyses as part of quality control work will therefore provide useful supplementary information to chemical analyses.

At present, there are no set of standards or requirements for the sensory quality of marine oils. On the other hand, in the field of plant oils there are a range of standards for both chemical and sensory quality control and standardization purposes (AOCS 2003, CODEX:19 2009a, CODEX:33 2009b, USDA 2010, CODEX:210 2011, USDA 2012, CODEX:132303 2013). In the case of olive oil, a commercially available aroma wheel has been developed that describes positive and negative aromas, appearances and mouthfeel (Mojet and de Jong 1994, Gawel and Rogers 2009). This type of profiling is also used to describe quality, and is a basis for information to the consumer (Monteleone et al. 1996, Caporale et al. 2006). It gives information about storage stability (Monteleone et al. 1995) and a correlation with volatile components (Morales et al. 1995, Aparicio et al. 1996). Olive oil producers are required by the IOC (International Olive Council) to use sensory quality controls alongside chemical analyses in order to provide consumers with a taste guarantee (Monteleone and Langstaff 2014).

A group of companies have together with Møreforsking and Nofima developed a method for quality control of marine oils. This quality control test is the first step in the systematization of the sensory work that takes places in omega-3 companies (NMKL:201 2017). The method includes a preliminary sensory wheel and nomenclature based on various sensory characteristics (deviations) that can be found in marine oils. The sensory wheel has been published (Larssen et

al. 2018). The quality control method and the sensory wheel are the first steps towards a common industry standard in the field.

## 1.1 OBJECTIVE

The objective of this study is to develop a common Norwegian sensory industry standard (gold standard) for marine oils. This standard should correlate with previously established international chemical quality standards.

**Sub-objectives:**

- Describe sensory characteristics of marine oils and study correlations between sensory characteristics and the quality of raw materials, chemical oxidation parameters and fatty acid profiles.
- Further develop and establish a detailed nomenclature lexicon and aroma wheel for use in internal quality control work, which describe sensory deviations in marine oils. Differentiation between deviations that can be accepted and deviations that downgrade oils will be emphasized.
- Identify market requirements and customer acceptance in relation to sensory quality of marine oils.
- Competency development through obtaining knowledge and experience from the olive oil industry about the use of sensory specifications of requirements.
- Dissemination of the findings through scientific and popular scientific channels.

A delimitation of the objectives was done focusing on fish oils and triglycerides.

# 2. MATERIAL AND METHODS

## 2.1 COLLECTION OF FISH OILS

Forty-six oils representing the most common products delivered from the marine oil industry were collected from eight omega-3 producers (Table 1 and 2). The selection included oils from cod and pelagic species like anchovies and tuna, and tocopherol was added as antioxidant. All the oils were triglycerides. Oils were collected from the producers' daily production line in two rounds (trial 1 and trial 2) over a nine-month period. They were produced under normal industrial processing conditions and were all newly refined. The oils were labelled according to species, EPA- and DHA-concentration[1], and antioxidant. None of the oils had aromas added to them, but ten of the oils were collected before deodorization. These oils are only reported according to their volatile properties in chapter 3.6. The oils were bottled in 300 ml aluminium containers under a nitrogen blanket and stored at -20 °C until further analysis.

In addition, a selection of four common cod-liver oils and four EPA concentrates went through an accelerated oxidation process (trial 3). 300 ml containers were filled with 250 ml to ensure equal surface area (oil/air). The containers were stored at 20 °C without lids to allow contact with air. All the bottles were shaken/swirled daily to mix the oil with air. At day zero, 7, 14 and 20 the oils were analysed for sensory quality, primary and secondary oxidation parameters and volatile compounds.

[1] EPA (Eicosapentaenoic acid) and DHA (Docosahexaenoic acid) are recognized as the two most important omega-3 fatty acids. The omega-3 industry concentrates these two fatty acids in their omega-3 products to maximize health effects.

**Table 1. Fish oils collected for trial 1 and 2 and analysed for sensory and chemical parameters. Oils in *italics* have not been deodorized. Eight of the oils in trial 2 also went through accelerated storage (trial 3).**

| | Code | Collected | Main raw material | Composition | Chemical analysis | Sensory profiling (*scan) |
|---|---|---|---|---|---|---|
| **TRIAL 1** | 1CN_17 | 2017 | Cod liver | Natural | x | x |
| | 3AEC_17 | 2017 | Anchoveta | EPA concentrate | x | x |
| | 4AEC_17 | 2017 | Anchoveta | EPA concentrate | x | x* |
| | 5ADC_17 | 2017 | Anchoveta | DHA concentrate | x | x |
| | 6ADC_17 | 2017 | Anchoveta | DHA concentrate | x | x |
| | 7CN_17 | 2017 | Cod liver | Natural | x | x* |
| | 8CC_17 | 2017 | Cod liver | Concentrate | x | x |
| | 12ADC_17 | 2017 | Anchoveta | DHA concentrate | x | x |
| | *13ADC_17* | *2017* | *Anchoveta* | *DHA concentrate* | *x* | *x* |
| | 15CN_17 | 2017 | Cod liver | Natural | x | x* |
| | 16CN_17 | 2017 | Cod liver | Natural | x | x |
| | 17AEC_17 | 2017 | Anchoveta | EPA concentrate | x | x |
| | 18CN_17 | 2017 | Cod liver | Natural | x | x* |
| | 19CN_17 | 2017 | Cod liver | Natural | x | x* |
| | 20AN_17 | 2017 | Anchoveta | Natural | x | x |
| | 21AEC_17 | 2017 | Anchoveta | EPA concentrate | x | x* |
| | 22AEC_17 | 2017 | Anchoveta | EPA concentrate | x | x |
| | 23AEC_17 | 2017 | Anchoveta | EPA concentrate | x | x |
| | *101CN_17* | *2017* | *Cod liver* | *Natural* | *x* | *x* |
| | *104AEC_17* | *2017* | *Anchoveta* | *EPA concentrate* | *x* | *x** |
| | *105ADC_17* | *2017* | *Anchoveta* | *DHA concentrate* | *x* | *x** |
| | *106ADC_17* | *2017* | *Anchoveta* | *DHA concentrate* | *x* | *x** |
| | *107CN_17* | *2017* | *Cod liver* | *Natural* | *x* | *x** |
| | *116CN_17* | *2017* | *Cod liver* | *Natural* | *x* | *x* |
| | *117AEC_17* | *2017* | *Anchoveta* | *EPA concentrate* | *x* | *x** |
| | *120AN_17* | *2017* | *Anchoveta* | *Natural* | *x* | *x* |
| | *121AEC_17* | *2017* | *Anchoveta* | *EPA concentrate* | *x* | *x** |
| | *122AEC_17* | *2017* | *Anchoveta* | *EPA concentrate* | *x* | *x* |

**Table 2. Fish oils collected for trial 2 and analysed for sensory and chemically parameters. 8 of the oils also went through accelerated storage (trail 3).**

| | Code | Collected | Main raw material | Composition | Chemical analysis | Sensory profiling | Accelerated storage TRIAL 3 |
|---|---|---|---|---|---|---|---|
| TRIAL 2 | 1AN_18 | 2018 | Anchoveta | Natural | x | x | |
| | 2AEC_18 | 2018 | Anchoveta | EPA concentrate | x | x | |
| | 3AEC_18 | 2018 | Anchoveta | EPA concentrate | x | x | x |
| | 4TDC_18 | 2018 | Tuna | DHA concentrate | x | x | |
| | 5AEC_18 | 2018 | Anchoveta | EPA concentrate | x | x | x |
| | 6ADC_18 | 2018 | Anchoveta | DHA concentrate | x | x | |
| | 7CN_18 | 2018 | Cod liver | Natural | x | x | x |
| | 8AEC_18 | 2018 | Anchoveta | EPA concentrate | x | x | x |
| | 9AEC_18 | 2018 | Anchoveta | EPA concentrate | x | x | |
| | 10ADC_18 | 2018 | Anchoveta | DHA concentrate | x | x | |
| | 11AEC_18 | 2018 | Anchoveta | EPA concentrate | x | x | x |
| | 12CN_18 | 2018 | Cod liver | Natural | x | x | |
| | 13CN_18 | 2018 | Cod liver | Natural | x | x | x |
| | 14AEC_18 | 2018 | Anchoveta | EPA concentrate | x | x | |
| | 15CN_18 | 2018 | Cod liver | Natural | x | x | x |
| | 16CN_18 | 2018 | Cod liver | Natural | x | x | x |
| | 17AEC_18 | 2018 | Anchoveta | EPA concentrate | x | x | |
| | 18ADC_18 | 2018 | Anchoveta | DHA concentrate | x | x | |

## 2.2 FATTY ACID COMPOSITION

The fatty acid composition was determined according to AOCS Official Method Ce 1b-89 (AOCS 2009). The fatty acid methyl esters (FAME) were analysed using a Clarus 500 gas chromatograph (GC) with FID detector (Perkin Elmer, Shelton, CT, USA) with a Carbowax 20M column (25 m, 0.25 mm ID, 0.20 µm film thickness, Quadrex Corporation, Woodbridge, CT, USA). The fatty acids were identified by comparing retention times with the retention times in FAME standards and cod liver oil. A sample was methylated once, before running in duplicate on the GC.

## 2.3 SENSORY ANALYSIS

The selected oils were evaluated by a highly trained panel of 10 assessors (10 women, aged 37–64 years) at Nofima, who performed a sensory descriptive analysis according to *Generic Descriptive Analysis* as described by Lawless and Heymann (2010) and ISO 13229 Sensory analysis – Methodology – General guidance for establishing a sensory profile (2016). The assessors were tested, selected and trained according to ISO standards (ISO8586 2012), and the sensory laboratory follows the ISO standards (ISO 8589, 2007). The sensory panel had extensive experience with descriptive analysis of a wide range of products.

During the attribute generation phase the assessors developed a vocabulary describing samples, and they agreed upon a list of 22 characteristics in total (Table 3). No attribute describing the appearance of the oil was included. In a pre-test session, as described in Lawless and Heymann (2010), the judges were trained in the definition of the characteristics by testing samples that were considered extreme with respect to selected characteristics typical for fish oil.

**Table 3. Vocabulary describing odour (O), flavour (F), taste (T) and mouthfeel (M) of fish oils.**

| Characteristic | Definition | Keywords |
|---|---|---|
| Sourness (O+F) | Related to a fresh odour and flavour due to organic acids. | Citrus and green apple |
| Bitter (T) | Related to a bitter taste (caffeine or quinine). | Grapefruit and caffeine |
| Butter (O+F) | Related to a smooth, full flavour and odour of dairy butter. | Clarified butter and popcorn |
| Chemical (O+F) | Related to the odour and taste of chemicals. | Glue, plastic, synthetic and artificial |
| Fermented (O+F) | Related to the odour and taste of matured fish. | Dried and matured fish |
| Fish (O+F) | Related to the odour and taste of fresh fish. | Fresh sea, seaweed, mackerel and shellfish |
| Fruit (O) | Related to a sweet, overripe odour of fruit. | Melon, banana, ripe apple, sweet alcohol |
| Grassy (O+F) | Related to the taste of fresh grass. | Fresh grass and green tomato |
| Medicine (O+F) | Related to the odour and flavour of medicine. | Pharmacy, dental offices, ethanol and soap |
| Metal (O) | Related to the odour of iron sulphide ($FeSO_4$). | Metal shavings, iron and blood |

*Table 3 continues.*

| | | |
|---|---|---|
| Nut/seed (O) | Related to the odour of fresh nuts and seeds. | Fresh nuts, almonds and linseed |
| Process (O+F) | Related to the odour and flavour of the refining process. | Diesel, motor oil, burned oil, tar and clay |
| Pungent (M) | Related to a stinging, hawking, coughing feeling. | Chemical irritation (hark, prickly, cough) |
| Rancid (O+F) | Related to the odour and flavour of oxidized fats. | Paint, linseed oil and wax. |

Samples were presented to the assessor in 70 ml cups with a lid, containing 20 ml of oil at 20 °C. A continuous, non-structured scale was used for evaluation. The left side of the scale corresponded to the lowest intensity of each attribute (value 1.0) and the right side corresponded to the highest intensity (value 9.0). Each assessor did a monadic evaluation of the samples in two replicates at individual speed on a computerized system for direct recording of data (EyeQuestion, Software Logic8 BV, Utrecht, The Netherlands). Samples and replicates were served in a randomized order.

During the evaluation, the assessors were instructed to lift the lid off the sample and smell the sample before tasting. The panel was asked to rinse their palates between the samples with water (37 °C), and, if necessary, using cucumber or bread.

The sensory profiling was done during two periods of 3 days, with a total of 4–5 sessions each day with three samples in each session. Between sessions the panellists had a 15-minute break, and after three sessions the panel had a 1.5-hour break.

In addition, analysis of oils that had been exposed to storage was carried out.

## 2.4 PRIMARY AND SECONDARY OXIDATION

The twenty oils from the sensory profiling were analysed for oxidation parameters (shown in Table 1). Primary and secondary fat oxidation in the samples were determined by analysing the peroxide-, anisidine- and free fatty acid values. Oils were analysed with regard to free fatty acid (FFA) content and determined according to IUPAC (Method no. 2.201 1987). Results are expressed as g FFA 100 $g^{-1}$ lipids. The peroxide value was determined according to AOCS (1997). Results were expressed as meq peroxide $kg^{-1}$ lipids. The anisidine value was determined according to AOCS (2003).

## 2.5 COLOUR AND CONJUGATED DIENES (ABSORBANCE)

The colour of the oils was measured according to AOCS (AOCS 2009). The colour of the oil samples was categorized according to Lovibond Gardner colour scale 1 to 9, Disc 4/30 (The

Tintometer Ltd, UK) in a Lovibond Comperator 2000+ with Lovibond daylight 2000 unit (The Tintometer Ltd, UK).

The absorbance at 233 nm in oil is a measurement of the content of conjugated dienes. The conjugated dienes are a measurement of the oxidation status. Approximately 0.3000 g oil was accurately weighted in a 50 ml measuring flask and diluted with isooctane. One ml of this solution was further diluted in a 25m measuring flask. The absorbance at 233 mn was measured in a 10 mm quartz cuvette in a Shimadzu UVmini-1240 spectrophotometer. The absorbance value was corrected for the sample weight. $ABS_{Corrected}$= ($ABS_{Measured}$*0.3)/sample weight. The amount of conjugated dienes was calculated by the formula % dienes = (( $ABS_{Measured}$*1.25)/sample weight)-0.07)*0.91.

## 2.6 VOLATILES

Gas chromatography mass spectrometry (GC-MS) was performed as described by Olsen et al. (2005) with minor modifications: 5 g oil sample was distributed as evenly as possible in 250 ml Erlenmeyer flasks and a solution of ethyl heptanoate (>99 %, Sigma-Aldrich Chemie GmbH, Steinheim, Germany) in methanol (p.a., Merck GmbH, Darmstadt, Germany) was added as an internal standard. The samples were heated to 70 °C in a water bath and purged with 100 mL/min nitrogen through a Drechsel-head for 20 minutes. Volatiles were trapped on Tenax GR (mesh size 60/80, Alltech Associates Inc., Deerfield, IL, USA). Trapped compounds were desorbed at 250 °C for 5 minutes in a Markes Unity/Ultra automatic thermal desorption unit (Markes International ltd, Llantisant, England) and transferred to an Agilent 6890 GC System (Agilent, Palo Alto, CA, USA) with an Agilent 5973 Mass selective detector (a quadrupole) operated in electron impact (EI) ionization mode at 70 eV. The compounds were separated on a DB-WAXetr GC column from J&W Scientific/Agilent (0.25 mm i.d., 0.5 µm film, 30 m). Helium (99.9999 %) was used as carrier gas.

The Integration of peaks and tentative identification of compounds were performed with HP Chemstation (G1701CA version C.00.00, Agilent Technologies), NIST98 Mass Spectral Library (version 2, 2005, US Secretary of Commerce/Agilent). Identification of the components was confirmed by comparison of retention times and mass spectra of the sample peaks with those of pure standards. The concentration of the individual volatiles was calculated as ng per gram sample based on internal standard added prior to headspace gas sampling. The analysis was performed in duplicate for all samples. System performance was checked with blanks and standard samples before, during and after the sample series.

## 2.7 MARKET REQUIREMENTS

To get background information regarding development of the sensory standard we conducted interviews with and a survey of the producers of fish oils and some of their customers.

### 2.7.1 INTERVIEW OF PRODUCERS OF MARINE OILS

An interview template was constructed. Main topics were:

- production and market segments
- use of sensory methods internally in the company
- use of sensory wheel
- customer requirements regarding sensory characteristics
- benefits from having a sensory quality standard

Ten interviews with the following production companies were conducted:

- Marine Ingredients
- Pharma Marine
- Nordic Pharma
- Epax
- Berg LipidTech
- Vitux/Concordix
- Vesteraalens
- G.C. Riber oils
- Calanus
- Orkla Health

### 2.7.2 SURVEY AND INTERVIEW WITH BUYERS OF MARINE OIL PRODUCTS

An interview template was constructed. Main topics were:

- production and market segments
- use of sensory methods internally in the company
- use of vocabulary regarding odour and flavour
- customer requirements regarding sensory characteristics
- importance of a sensory quality standard

The survey was sent to eight customers of the marine oil producers.

## 2.8 CLASSIFICATION AND STANDARDIZATION

Three classifications of fish oils were suggested. First a selection of prominent sensory characteristics was made based on the sensory and chemical analysis of a total of 70 oils, feedback from the market, and discussion with the omega-3 industry. The discussions initially used the 21 characteristics and 60 keywords described by Larssen et al. (2018). Second, an intensity scale of the selected characteristics was made that allowed a certain intensity of some of the characteristics in the classifications.

Using the three classifications, an adjustment of the scaling in NMKL:201 (2017) was suggested in order to use this quality control method in the standardization process.

Re-classification of the oils after the sensory profiling was done to study differences between the classifications and the oxidation parameters. The 9-point intensity scale used in the sensory profiling was replaced by a 5-point scale (Table 4), and the results translated accordingly.

**Table 4. Correspondence between semantic intensity categories, a 5-point scale, and the continuous, non-structured 9-point rating scale used by the trained panel at NOFIMA.**

| Intensity | 5-point scale | 9-point scale |
|---|---|---|
| Very low | 0 | 1 ≤ Int.<2 |
| Low | 1 | 2 ≤ Int.<4 |
| Moderate | 2 | 4 ≤ Int.<6 |
| Strong | 3 | 6 ≤ Int.<8 |
| Very Strong | 4 | 8 ≤ Int. ≤ 9 |

int. = intensity

## 2.9 STATISTICS

Data from the sensory descriptive analysis were evaluated using analysis of variance (ANOVA). Least significant differences were calculated by Tukey's HSD test ($P<0.05$). The model terms product, replicate and interactions involving these terms were considered fixed, while the assessor and interaction effects including assessor were considered random. The analysis was performed on the descriptive sensory data from the trained panel in order to identify the sensory characteristics that discriminated between samples. A principal component analysis (PCA) on the average of the sensory descriptive data was done with mean centred data and no standardization.

The statistical software used for the sensory analysis was EyeOpenR (Logic8 BV). For the multivariate data analysis, Unscrambler X Version 10.4.1. was used for the PCA.

For the multivariate data analysis of sensory and chemical data, the software package The Unscrambler© (Version 9.8, Camo Norway) was used. Principal component analysis (PCA) was used to analyse the variance of sensory and chemical data, and for the correlation computations between sensory and chemical data partial least squares regression (PLSR) was used with segmented cross validation of replicates.

Statistical analyses on differences between classifications were performed in Graph pad Prism 8.0.0 (224). The data for total volatiles, PV, AnV, FFA and colour were log-transformed, and mean values were compared using one-way analysis of variance (ANOVA) with Tukey post hoc test. The mean values for volatile compounds were not normally distributed and were compared using Kruskal-Wallis test with Dunn's post hoc test.

# 3. RESULTS

## 3.1 FATTY ACID COMPOSITION

Oils collected from eight of the nine industry partners in the project all had different fatty acid compositions (areal %). The natural oil from cod liver had 8.3-10.3 EPA and 11.0-12.7 DHA (Table 5). The anchoveta oils are represented both as natural oils and concentrates. The highest registered EPA concentration in the sample set was 51.9 and the highest DHA concentration registered was 74.2.

**Table 5. Concentration of the two most important fatty acids (EPA and DHA) in the collected fish oils. Results are shown as averages between two measurements.**

| TRIAL 1 | | | TRIAL 2 | | |
|---|---|---|---|---|---|
| Code | C20:5(n-3) EPA (areal%) | C22:6(n-3) DHA (areal%) | Code | C20:5(n-3) EPA (areal%) | C22:6(n-3) DHA (areal%) |
| 1CN_17 | 9.5 | 11.1 | 1AN_18 | 20.5 | 13.7 |
| 3AEC_17 | 37.0 | 23.1 | 2AEC_18 | 45.0 | 22.2 |
| 4AEC_17 | 40.5 | 25.7 | 3AEC_18 | 36.0 | 25.7 |
| 5ADC_17 | 10.9 | 74.2 | 4TDC_18 | 12.1 | 56.6 |
| 6ADC_17 | 14.7 | 64.7 | 5AEC_18 | 36.4 | 24.2 |
| 7CN_17 | 10.0 | 11.2 | 6ADC_18 | 17.3 | 60.8 |
| 8CC_17 | 16.0 | 34.3 | 7CN_18 | 8.5 | 12.3 |
| 12ADC_17 | 8.4 | 67.2 | 8AEC_18 | 45.8 | 32.2 |
| *13ADC_17* | *16.9* | *52.5* | 9AEC_18 | 43.8 | 24.2 |
| 15CN_17 | 8.2 | 11.6 | 10ADC_18 | 51.5 | 19.0 |
| 16CN_17 | 8.6 | 11.0 | 11AEC_18 | 45.0 | 36.6 |
| 17AEC_17 | 36.8 | 23.1 | 12CN_18 | 8.8 | 12.7 |
| 18CN_17 | 8.6 | 11.0 | 13CN_18 | 8.2 | 12.4 |
| 19CN_17 | 8.7 | 11.4 | 14AEC_18 | 42.2 | 28.5 |
| 20AN_17 | 18.8 | 13.3 | 15CN_18 | 8.5 | 12.4 |
| 21AEC_17 | 36.8 | 24.4 | 16CN_18 | 8.4 | 12.6 |
| 22AEC_17 | 36.8 | 22.7 | 17AEC_18 | 35.3 | 23.5 |
| 23AEC_17 | 51.9 | 17.4 | 18ADC_18 | 14.0 | 60.3 |

Code: C = cod liver oil, A = anchoveta oil, N = natural oil, EC = EPA concentrate, DC = DHA concentrate

Table 6 shows the fatty acid composition of the un-deodorized samples collected during autumn 2017. Sample *101CN_17* is the same as 1CN_17 before deodorization, and, as Table 3 and 4

show, the deodorization process gives a slight reduction of the EPA and DHA fatty acid concentration in the samples. The reduction is less than 10 % for most of the samples. The exceptions are samples 17AEC_17 and 117AEC_17, where the samples had a higher fatty acid concentration after deodorization, which suggests that the samples were collected in early stages of the refining process before the fatty acids had been up-concentrated.

**Table 6. Concentration of the two most important fatty acids (EPA and DHA) in the collected fish oils that had been deodorized.**

| **TRIAL 1: not deodorized** | | |
|---|---|---|
| Code | C20:5(n-3) EPA (areal%) | C22:6(n-3) DHA (areal%) |
| *101CN_17* | *10.3* | *11.7* |
| *104AEC_17* | *40.2* | *26.0* |
| *105ADC_17* | *10.6* | *74.5* |
| *106ADC_17* | *15.4* | *63.1* |
| *107CN_17* | *10.2* | *11.9* |
| *116CN_17* | *8.9* | *11.7* |
| *117AEC_17* | *17.9* | *11.3* |
| *120AN_17* | *18.7* | *12.5* |
| *121AEC_17* | *37.8* | *23.7* |
| *122AEC_17* | *36.8* | *22.6* |

Code: C = cod liver oil, A = anchoveta oil, N = natural oil, EC = EPA concentrate, DC = DHA concentrate

## 3.2 SENSORY ANALYSIS

### 3.2.1 TRIAL 1

Results from 12 refined oils were analysed (Table 7 a and b). Among 22 tested sensory characteristics, 17 showed significant differences between the fish oils, table 7 ab.

**Table 7 a. Odour values for the fish oils in trial 1. Mean of x samples shown. Different letters indicate significant difference ($p<0.05$) among the fish oil products using two-way ANOVA and Tukey's multiple comparison test.**

| Sample | Sourness odour | Grassy odour | Fish odour | Butter odour | Metal odour | Fruit odour | Nut/seed odour | Chemical odour | Process odour | Medicine odour | Fermented odour | Rancid odour |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **1CN** | 2.38 $^{bc}$ | 2.36 $^{a}$ | 1.96 $^{b}$ | 1.07 $^{b}$ | 3.41 $^{abcd}$ | 1.92 $^{a}$ | 1.01 $^{a}$ | 3.59 $^{ab}$ | 2.51 $^{ab}$ | 1.58 $^{a}$ | 1.51 $^{ab}$ | 3.44 $^{ab}$ |
| **3AEC** | 1.43 $^{c}$ | 1.74 $^{a}$ | 1.96 $^{b}$ | 1.01 $^{b}$ | 3.74 $^{ab}$ | 1.28 $^{a}$ | 1.12 $^{a}$ | 2.69 $^{bc}$ | 3.52 $^{a}$ | 1.32 $^{a}$ | 2.22 $^{a}$ | 5.07 $^{a}$ |
| **5ADC** | 2.82 $^{abc}$ | 2.23 $^{a}$ | 1.93 $^{b}$ | 1.32 $^{b}$ | 3.33 $^{abcd}$ | 1.67 $^{a}$ | 1.03 $^{a}$ | 2.73 $^{bc}$ | 1.81 $^{b}$ | 1.52 $^{a}$ | 1.46 $^{ab}$ | 2.89 $^{abc}$ |
| **6ADC** | 2.77 $^{abc}$ | 2.01 $^{a}$ | 3.98 $^{a}$ | 1.04 $^{b}$ | 3.97 $^{a}$ | 1.23 $^{a}$ | 1.01 $^{a}$ | 1.28 $^{c}$ | 2.26 $^{ab}$ | 1.11 $^{a}$ | 1.53 $^{ab}$ | 3.06 $^{abc}$ |
| **8CC** | 3.63 $^{ab}$ | 2.58 $^{a}$ | 3.23 $^{ab}$ | 1.09 $^{b}$ | 3.26 $^{abcd}$ | 1.25 $^{a}$ | 1.08 $^{a}$ | 1.44 $^{c}$ | 1.56 $^{b}$ | 1.09 $^{a}$ | 1.43 $^{ab}$ | 2.12 $^{bc}$ |
| **12ADC** | 3.37 $^{ab}$ | 1.34 $^{a}$ | 2.29 $^{b}$ | 1.06 $^{b}$ | 2.22 $^{d}$ | 1.01 $^{a}$ | 1.01 $^{a}$ | 1.11 $^{c}$ | 1.01 $^{b}$ | 1.01 $^{a}$ | 1.29 $^{ab}$ | 1.14 $^{c}$ |
| **13ADC** | 4.27 $^{a}$ | 1.69 $^{a}$ | 2.27 $^{b}$ | 1.05 $^{b}$ | 2.18 $^{d}$ | 1.26 $^{a}$ | 1.02 $^{a}$ | 1.04 $^{c}$ | 1.01 $^{b}$ | 1.14 $^{a}$ | 1.01 $^{b}$ | 1.18 $^{c}$ |
| **16CN** | 3.26 $^{ab}$ | 1.64 $^{a}$ | 3.03 $^{ab}$ | 1.33 $^{b}$ | 3.29 $^{abcd}$ | 1.08 $^{a}$ | 1.37 $^{a}$ | 1.67 $^{bc}$ | 1.40 $^{b}$ | 1.01 $^{a}$ | 1.15 $^{ab}$ | 2.62 $^{bc}$ |
| **18CN** | 3.99 $^{a}$ | 2.29 $^{a}$ | 2.20 $^{b}$ | 2.27 $^{a}$ | 2.47 $^{cd}$ | 1.84 $^{a}$ | 1.18 $^{a}$ | 1.83 $^{bc}$ | 1.28 $^{b}$ | 1.01 $^{a}$ | 1.11 $^{ab}$ | 1.57 $^{bc}$ |
| **20AN** | 3.56 $^{ab}$ | 1.51 $^{a}$ | 2.39 $^{b}$ | 1.07 $^{b}$ | 2.51 $^{bcd}$ | 1.01 $^{a}$ | 1.20 $^{a}$ | 1.16 $^{c}$ | 1.07 $^{b}$ | 1.09 $^{a}$ | 1.01 $^{b}$ | 1.57 $^{bc}$ |
| **22AEC** | 2.39 $^{bc}$ | 2.02 $^{a}$ | 2.38 $^{b}$ | 1.09 $^{b}$ | 3.53 $^{abc}$ | 1.42 $^{a}$ | 1.42 $^{a}$ | 2.27 $^{bc}$ | 2.02 $^{ab}$ | 1.13 $^{a}$ | 1.56 $^{ab}$ | 3.66 $^{ab}$ |
| **23AEC** | 2.37 $^{bc}$ | 1.87 $^{a}$ | 2.54 $^{ab}$ | 1.13 $^{b}$ | 3.30 $^{abcd}$ | 1.53 $^{a}$ | 1.07 $^{a}$ | 4.78 $^{a}$ | 2.12 $^{ab}$ | 1.86 $^{a}$ | 1.84 $^{ab}$ | 2.97 $^{abc}$ |
| **p-value** | <0.001 | 0.147 | <0.001 | <0.001 | <0.001 | 0.007 | 0.276 | <0.001 | <0.001 | 0.237 | 0.032 | <0.001 |

**Table 7 b. Flavour, taste and mouthfeel values for the fish oils in trial 1. Mean of x samples shown. Different letters indicate significant difference (p<0.05) among the fish oil products using two-way ANOVA and Tukey's multiple comparison test.**

| Sample | Sourness flavour | Bitter taste | Grassy flavour | Fish flavour | Butter flavour | Chemical flavour | Process flavour | Fermented flavour | Rancid flavour | Pungent |
|---|---|---|---|---|---|---|---|---|---|---|
| **1CN** | 2.17 $^{cd}$ | 4.20 $^{ab}$ | 2.19 $^{a}$ | 2.38 $^{b}$ | 1.05 $^{a}$ | 2.87 $^{ab}$ | 2.92 $^{ab}$ | 2.50 $^{ab}$ | 3.52 $^{abc}$ | 3.97 $^{abc}$ |
| **3AEC** | 1.21 $^{d}$ | 4.59 $^{a}$ | 1.34 $^{a}$ | 2.07 $^{b}$ | 1.01 $^{a}$ | 2.93 $^{ab}$ | 4.06 $^{a}$ | 3.59 $^{a}$ | 5.29 $^{a}$ | 4.16 $^{ab}$ |
| **5ADC** | 2.63 $^{bcd}$ | 3.65 $^{abcd}$ | 1.83 $^{a}$ | 2.18 $^{b}$ | 1.13 $^{a}$ | 2.64 $^{ab}$ | 1.86 $^{bc}$ | 1.39 $^{bc}$ | 3.17 $^{bcd}$ | 2.77 $^{cd}$ |
| **6ADC** | 2.90 $^{bc}$ | 3.93 $^{abcd}$ | 2.09 $^{a}$ | 4.59 $^{a}$ | 1.01 $^{a}$ | 1.37 $^{b}$ | 2.18 $^{bc}$ | 2.03 $^{bc}$ | 3.13 $^{bcd}$ | 3.25 $^{bcd}$ |
| **8CC** | 3.94 $^{ab}$ | 3.69 $^{abcd}$ | 2.65 $^{a}$ | 3.71 $^{ab}$ | 1.15 $^{a}$ | 1.42 $^{b}$ | 1.78 $^{bc}$ | 1.38 $^{bc}$ | 2.46 $^{bcd}$ | 3.19 $^{bcd}$ |
| **12ADC** | 3.83 $^{ab}$ | 3.21 $^{cd}$ | 1.68 $^{a}$ | 1.85 $^{b}$ | 1.20 $^{a}$ | 1.33 $^{b}$ | 1.21 $^{c}$ | 1.12 $^{c}$ | 1.33 $^{d}$ | 2.12 $^{d}$ |
| **13ADC** | 3.81 $^{ab}$ | 2.99 $^{d}$ | 1.77 $^{a}$ | 2.82 $^{ab}$ | 1.19 $^{a}$ | 1.67 $^{ab}$ | 1.79 $^{bc}$ | 1.19 $^{c}$ | 1.52 $^{d}$ | 2.27 $^{d}$ |
| **16CN** | 2.96 $^{abc}$ | 3.69 $^{abcd}$ | 1.93 $^{a}$ | 2.75 $^{ab}$ | 1.30 $^{a}$ | 2.49 $^{ab}$ | 1.53 $^{bc}$ | 1.36 $^{bc}$ | 2.97 $^{bcd}$ | 2.52 $^{d}$ |
| **18CN** | 4.44 $^{a}$ | 3.55 $^{bcd}$ | 2.54 $^{a}$ | 3.04 $^{ab}$ | 1.33 $^{a}$ | 1.44 $^{b}$ | 1.47 $^{bc}$ | 1.17 $^{c}$ | 2.13 $^{bcd}$ | 2.69 $^{cd}$ |
| **20AN** | 3.98 $^{ab}$ | 3.22 $^{bcd}$ | 2.08 $^{a}$ | 2.33 $^{b}$ | 1.09 $^{a}$ | 1.38 $^{b}$ | 1.41 $^{bc}$ | 1.33 $^{bc}$ | 1.79 $^{cd}$ | 2.23 $^{d}$ |
| **22AEC** | 2.48 $^{bcd}$ | 4.03 $^{abc}$ | 2.28 $^{a}$ | 2.91 $^{ab}$ | 1.13 $^{a}$ | 2.05 $^{ab}$ | 2.14 $^{bc}$ | 1.61 $^{bc}$ | 3.42 $^{abc}$ | 3.04 $^{bcd}$ |
| **23AEC** | 2.62 $^{bcd}$ | 4.57 $^{a}$ | 1.88 $^{a}$ | 3.02 $^{ab}$ | 1.06 $^{a}$ | 3.93 $^{a}$ | 2.32 $^{bc}$ | 1.92 $^{bc}$ | 3.78 $^{ab}$ | 4.59 $^{a}$ |
| **p-value** | <0.001 | <0.001 | 0.072 | <0.001 | 0.153 | 0.001 | <0.002 | <0.001 | <0.001 | <0.001 |

Principal component analysis (PCA) was used to find similarities and differences between the sensory characteristics and the fish oils (Figure 1). The first and second principal components (PCs) explained 70 % and 12 % of the plot, respectively, which means the first PCs dominate the interpretation of the plots moving in the direction from *sourness* to *rancid*.

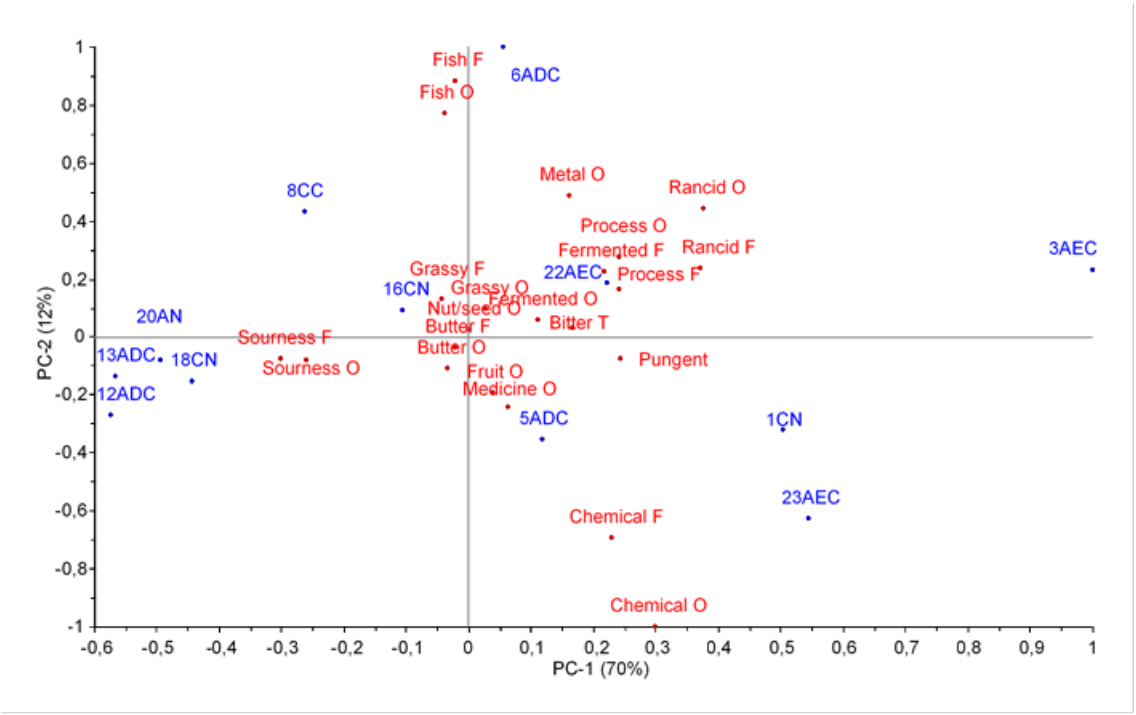


**Figure 1. Principal component analysis (PCA) bi-plot showing similarities and differences between sensory characteristics and fish oils in trial 1.**

Overall, going from left to right in the figure, one can see the characteristics describing and differentiating the fish oils in the order *sourness-grassy-bitter-process-rancid*. Fish oils 12ADC, 13ADC, 20AN, 18CN and 8CC are grouped and described as having *sourness* odour and flavour, while fish oils 3AEC, 1CN and 23AEC are grouped and described as having *rancid*, *process*, *fermented* odour and flavour and *bitter* taste. The second principal component is somehow differentiating fish oil 6ADC by having more fish odour and flavour, while on the opposite side fish oil 23AEC is differentiated as having a more *chemical* odour and flavour.

When considering the use of different raw materials and the different concentrations of EPA and DHA in the fish oils, no correlations are found. Anchoveta oil and cod liver oil are spread all over the figure, and the same is found for natural oils vs. concentrates. There is a trend towards EPA concentrates to be described as more rancid compared to the DHA concentrates.

### 3.2.2 TRIAL 2

Results from sensory analyses of 18 refined oils are shown in table 8 a and b.

**Table 8 a. Odour values for the fish oils in trial 2. Mean of x samples shown. Different letters indicate significant difference (p<0.05) among the fish oil products using two-way ANOVA and Tukey's multiple comparison test.**

| Sample | Sourness odour | Grassy odour | Fish odour | Butter odour | Metal odour | Fruit odour | Nut/seed odour | Chemical odour | Process odour | Medicine odour | Fermented odour | Rancid odour |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1AN | 3.44 $^{ab}$ | 1.44 $^{ab}$ | 2.12 $^{a}$ | 1.12 $^{a}$ | 2.32 $^{cd}$ | 1.01 $^{b}$ | 1.10 $^{a}$ | 1.40 $^{ab}$ | 1.29 $^{fg}$ | 1.01 $^{b}$ | 1.09 $^{ef}$ | 1.22 $^{f}$ |
| 2AEC | 1.56 $^{de}$ | 1.84 $^{ab}$ | 2.05 $^{a}$ | 1.07 $^{a}$ | 4.41 $^{ab}$ | 1.62 $^{ab}$ | 1.13 $^{a}$ | 2.86 $^{ab}$ | 4.33 $^{abc}$ | 1.41 $^{ab}$ | 3.04 $^{bcdef}$ | 4.39 $^{bc}$ |
| 3AEC | 2.19 $^{abcde}$ | 1.81 $^{ab}$ | 1.74 $^{a}$ | 1.09 $^{a}$ | 4.17 $^{ab}$ | 1.04 $^{b}$ | 1.04 $^{a}$ | 2.99 $^{ab}$ | 3.93 $^{bcd}$ | 1.23 $^{b}$ | 3.41 $^{bcde}$ | 4.13 $^{bcd}$ |
| 4TDC | 3.74 $^{a}$ | 2.06 $^{ab}$ | 2.12 $^{a}$ | 1.14 $^{a}$ | 2.14 $^{cd}$ | 1.32 $^{ab}$ | 1.45 $^{a}$ | 1.29 $^{ab}$ | 1.13 $^{g}$ | 1.04 $^{b}$ | 1.01 $^{f}$ | 1.04 $^{f}$ |
| 5AEC | 2.40 $^{abcde}$ | 2.61 $^{ab}$ | 2.33 $^{a}$ | 1.02 $^{a}$ | 4.26 $^{ab}$ | 1.21 $^{ab}$ | 1.26 $^{a}$ | 2.46 $^{ab}$ | 3.62 $^{bcde}$ | 1.35 $^{ab}$ | 2.54 $^{bcdef}$ | 3.89 $^{bcde}$ |
| 6ADC | 2.43 $^{abcde}$ | 2.59 $^{ab}$ | 1.89 $^{a}$ | 1.14 $^{a}$ | 3.50 $^{abc}$ | 2.29 $^{a}$ | 1.33 $^{a}$ | 3.47 $^{a}$ | 3.35 $^{bcdef}$ | 1.11 $^{b}$ | 2.75 $^{bcdef}$ | 2.24 $^{def}$ |
| 7CN | 3.58 $^{a}$ | 2.86 $^{a}$ | 2.51 $^{a}$ | 1.04 $^{a}$ | 3.06 $^{bcd}$ | 1.44 $^{ab}$ | 1.19 $^{a}$ | 1.33 $^{ab}$ | 1.97 $^{defg}$ | 1.01 $^{b}$ | 1.70 $^{cdef}$ | 1.66 $^{f}$ |
| 8AEC | 1.23 $^{e}$ | 1.15 $^{b}$ | 1.31 $^{a}$ | 1.03 $^{a}$ | 3.53 $^{abc}$ | 1.16 $^{ab}$ | 1.01 $^{a}$ | 1.77 $^{ab}$ | 1.65 $^{efg}$ | 1.15 $^{b}$ | 5.93 $^{a}$ | 2.62 $^{cdef}$ |
| 9AEC | 3.61 $^{a}$ | 2.26 $^{ab}$ | 2.25 $^{a}$ | 1.04 $^{a}$ | 2.25 $^{cd}$ | 1.31 $^{ab}$ | 1.10 $^{a}$ | 1.14 $^{b}$ | 1.16 $^{g}$ | 1.01 $^{b}$ | 1.01 $^{f}$ | 1.11 $^{f}$ |
| 10ADC | 1.54 $^{de}$ | 2.13 $^{ab}$ | 2.09 $^{a}$ | 1.07 $^{a}$ | 4.66 $^{a}$ | 1.15 $^{b}$ | 1.12 $^{a}$ | 3.41 $^{ab}$ | 4.67 $^{ab}$ | 1.64 $^{ab}$ | 3.44 $^{bcd}$ | 4.57 $^{bc}$ |
| 11AEC | 3.35 $^{abc}$ | 2.01 $^{ab}$ | 1.67 $^{a}$ | 1.01 $^{a}$ | 2.30 $^{cd}$ | 1.41 $^{ab}$ | 1.11 $^{a}$ | 1.27 $^{ab}$ | 1.17 $^{g}$ | 1.41 $^{ab}$ | 1.10 $^{ef}$ | 1.24 $^{f}$ |
| 12CN | 3.49 $^{ab}$ | 2.48 $^{ab}$ | 2.26 $^{a}$ | 1.17 $^{a}$ | 3.48 $^{abc}$ | 1.16 $^{ab}$ | 1.46 $^{a}$ | 1.75 $^{ab}$ | 1.92 $^{defg}$ | 1.09 $^{b}$ | 1.67 $^{def}$ | 2.01 $^{ef}$ |
| 13CN | 1.54 $^{de}$ | 1.66 $^{ab}$ | 2.03 $^{a}$ | 1.04 $^{a}$ | 4.26 $^{ab}$ | 1.39 $^{ab}$ | 1.23 $^{a}$ | 2.20 $^{ab}$ | 4.23 $^{abc}$ | 1.31 $^{ab}$ | 4.01 $^{abc}$ | 4.77 $^{ab}$ |
| 14AEC | 1.48 $^{e}$ | 1.44 $^{ab}$ | 1.70 $^{a}$ | 1.10 $^{a}$ | 4.46 $^{ab}$ | 1.01 $^{b}$ | 1.11 $^{a}$ | 2.48 $^{ab}$ | 3.27 $^{bcdef}$ | 1.01 $^{b}$ | 4.65 $^{ab}$ | 3.83 $^{bcde}$ |
| 15CN | 1.80 $^{cde}$ | 1.35 $^{ab}$ | 1.55 $^{a}$ | 1.21 $^{a}$ | 3.40 $^{abc}$ | 1.27 $^{ab}$ | 1.01 $^{a}$ | 2.20 $^{ab}$ | 2.28 $^{cdefg}$ | 1.37 $^{ab}$ | 3.23 $^{bcdef}$ | 2.04 $^{ef}$ |
| 16CN | 1.95 $^{bcde}$ | 1.94 $^{ab}$ | 2.30 $^{a}$ | 1.14 $^{a}$ | 4.11 $^{ab}$ | 1.21 $^{ab}$ | 1.18 $^{a}$ | 2.77 $^{ab}$ | 3.12 $^{bcdefg}$ | 1.12 $^{b}$ | 2.49 $^{bcdef}$ | 3.03 $^{bcdef}$ |
| 17AEC | 1.03 $^{e}$ | 1.48 $^{ab}$ | 1.12 $^{a}$ | 1.12 $^{a}$ | 4.09 $^{ab}$ | 1.70 $^{ab}$ | 1.29 $^{a}$ | 3.24 $^{ab}$ | 6.21 $^{a}$ | 2.36 $^{a}$ | 4.74 $^{ab}$ | 6.81 $^{a}$ |
| 18ADC | 3.11 $^{abcd}$ | 1.74 $^{ab}$ | 2.24 $^{a}$ | 1.06 $^{a}$ | 1.91 $^{d}$ | 1.32 $^{ab}$ | 1.10 $^{a}$ | 1.44 $^{ab}$ | 1.17 $^{g}$ | 1.07 $^{b}$ | 1.34 $^{def}$ | 1.43 $^{f}$ |
| p-value | <0.001 | 0.01 | 0.321 | 0.648 | <0.001 | 0.033 | 0.415 | <0.001 | <0.001 | 0.005 | <0.001 | <0.001 |

**Table 8 b. Flavour, taste and mouthfeel mean sensory intensity values for the fish oils in trial 2. Different letters indicate significant difference (p<0.05) among the fish oil products using two-way ANOVA and Tukey's multiple comparison test.**

| Sample | Sourness flavour | Bitter taste | Grassy flavour | Fish flavour | Butter flavour | Chemical flavour | Process flavour | Fermented flavour | Rancid flavour | Pungent |
|---|---|---|---|---|---|---|---|---|---|---|
| 1AN | 3.59 $^{abcd}$ | 3.16 $^{cde}$ | 2.36 $^{abcd}$ | 2.03 $^{a}$ | 1.31 $^{a}$ | 1.51 $^{a}$ | 1.71 $^{de}$ | 1.11 $^{d}$ | 1.32 $^{f}$ | 3.42 $^{bcdef}$ |
| 2AEC | 1.97 $^{efgh}$ | 4.49 $^{ab}$ | 2.51 $^{abcd}$ | 2.32 $^{a}$ | 1.09 $^{a}$ | 3.19 $^{a}$ | 4.18 $^{bc}$ | 3.23 $^{abcd}$ | 4.17 $^{abcd}$ | 4.41 $^{abcde}$ |
| 3AEC | 2.23 $^{cdefgh}$ | 4.27 $^{abc}$ | 2.46 $^{abcd}$ | 2.08 $^{a}$ | 1.14 $^{a}$ | 2.44 $^{a}$ | 3.78 $^{bcd}$ | 2.83 $^{abcd}$ | 3.48 $^{cde}$ | 4.11 $^{abcdef}$ |
| 4TDC | 3.92 $^{ab}$ | 2.70 $^{e}$ | 2.06 $^{abcd}$ | 2.12 $^{a}$ | 1.26 $^{a}$ | 1.62 $^{a}$ | 1.19 $^{e}$ | 1.07 $^{d}$ | 1.18 $^{f}$ | 2.61 $^{f}$ |
| 5AEC | 2.17 $^{defgh}$ | 4.53 $^{ab}$ | 2.22 $^{abcd}$ | 2.31 $^{a}$ | 1.17 $^{a}$ | 2.36 $^{a}$ | 4.18 $^{bc}$ | 2.81 $^{abcd}$ | 4.41 $^{abcd}$ | 4.68 $^{abcd}$ |
| 6ADC | 2.71 $^{bcdefg}$ | 4.27 $^{abc}$ | 3.23 $^{ab}$ | 1.83 $^{a}$ | 1.59 $^{a}$ | 3.27 $^{a}$ | 3.36 $^{bcd}$ | 2.14 $^{cd}$ | 2.00 $^{ef}$ | 4.33 $^{abcde}$ |
| 7CN | 3.47 $^{abcde}$ | 3.31 $^{cde}$ | 2.69 $^{abcd}$ | 2.84 $^{a}$ | 1.14 $^{a}$ | 1.53 $^{a}$ | 2.27 $^{cde}$ | 2.14 $^{cd}$ | 1.98 $^{ef}$ | 3.36 $^{cdef}$ |
| 8AEC | 1.59 $^{gh}$ | 4.19 $^{abc}$ | 1.70 $^{bcd}$ | 1.41 $^{a}$ | 1.23 $^{a}$ | 2.04 $^{a}$ | 2.69 $^{bcde}$ | 4.53 $^{ab}$ | 2.42 $^{def}$ | 4.26 $^{abcdef}$ |
| 9AEC | 4.94 $^{a}$ | 2.83 $^{de}$ | 3.55 $^{a}$ | 1.93 $^{a}$ | 1.19 $^{a}$ | 1.01 $^{a}$ | 1.04 $^{e}$ | 1.01 $^{d}$ | 1.01 $^{f}$ | 2.61 $^{f}$ |
| 10ADC | 1.76 $^{fgh}$ | 4.76 $^{ab}$ | 2.49 $^{abcd}$ | 2.20 $^{a}$ | 1.19 $^{a}$ | 3.11 $^{a}$ | 4.84 $^{ab}$ | 3.21 $^{abcd}$ | 5.08 $^{abc}$ | 5.08 $^{ab}$ |
| 11AEC | 3.80 $^{abc}$ | 3.17 $^{cde}$ | 2.38 $^{abcd}$ | 1.69 $^{a}$ | 1.18 $^{a}$ | 1.37 $^{a}$ | 1.11 $^{e}$ | 1.21 $^{d}$ | 1.05 $^{f}$ | 2.86 $^{ef}$ |
| 12CN | 3.21 $^{bcdef}$ | 3.97 $^{bcd}$ | 3.00 $^{abc}$ | 2.39 $^{a}$ | 1.35 $^{a}$ | 1.88 $^{a}$ | 2.77 $^{bcde}$ | 1.73 $^{cd}$ | 2.71 $^{def}$ | 3.54 $^{bcdef}$ |
| 13CN | 1.20 $^{gh}$ | 4.64 $^{ab}$ | 1.40 $^{cd}$ | 1.91 $^{a}$ | 1.03 $^{a}$ | 2.37 $^{a}$ | 4.83 $^{ab}$ | 4.62 $^{ab}$ | 5.53 $^{ab}$ | 4.92 $^{abc}$ |
| 14AEC | 1.52 $^{gh}$ | 4.11 $^{bc}$ | 1.56 $^{bcd}$ | 1.56 $^{a}$ | 1.25 $^{a}$ | 2.47 $^{a}$ | 4.01 $^{bc}$ | 4.00 $^{abc}$ | 4.34 $^{abcd}$ | 3.83 $^{bcdef}$ |
| 15CN | 1.72 $^{fgh}$ | 3.88 $^{bcd}$ | 1.56 $^{bcd}$ | 1.52 $^{a}$ | 1.41 $^{a}$ | 3.14 $^{a}$ | 2.54 $^{cde}$ | 3.86 $^{abc}$ | 2.56 $^{def}$ | 4.34 $^{abcde}$ |
| 16CN | 2.23 $^{cdefgh}$ | 3.92 $^{bcd}$ | 2.23 $^{abcd}$ | 2.17 $^{a}$ | 1.11 $^{a}$ | 2.73 $^{a}$ | 3.69 $^{bcd}$ | 2.42 $^{bcd}$ | 3.53 $^{bcde}$ | 4.01 $^{bcdef}$ |
| 17AEC | 1.02 $^{h}$ | 5.31 $^{a}$ | 1.32 $^{d}$ | 1.16 $^{a}$ | 1.07 $^{a}$ | 3.20 $^{a}$ | 6.81 $^{a}$ | 4.95 $^{a}$ | 6.01 $^{a}$ | 5.69 $^{a}$ |
| 18ADC | 3.65 $^{abcd}$ | 3.33 $^{cde}$ | 1.85 $^{bcd}$ | 1.94 $^{a}$ | 1.12 $^{a}$ | 1.66 $^{a}$ | 1.69 $^{de}$ | 1.36 $^{d}$ | 1.63 $^{ef}$ | 3.24 $^{def}$ |
| p-value | <0.001 | <0.001 | <0.001 | 0.172 | 0.341 | 0.003 | <0.001 | <0.001 | <0.001 | <0.001 |

From 22 tested sensory characteristics 17 showed significant differences between the fish oils (Table 8 a and b). Principal component analysis (PCA) was used to find similarities and differences between the sensory characteristics and the fish oils (Figure 2). The first and second principal components (PCs) explained 81 % and 9 % the plot, respectively, which means that the first PCs dominate the interpretation of the plots moving in the direction from *sourness* to *rancid*.

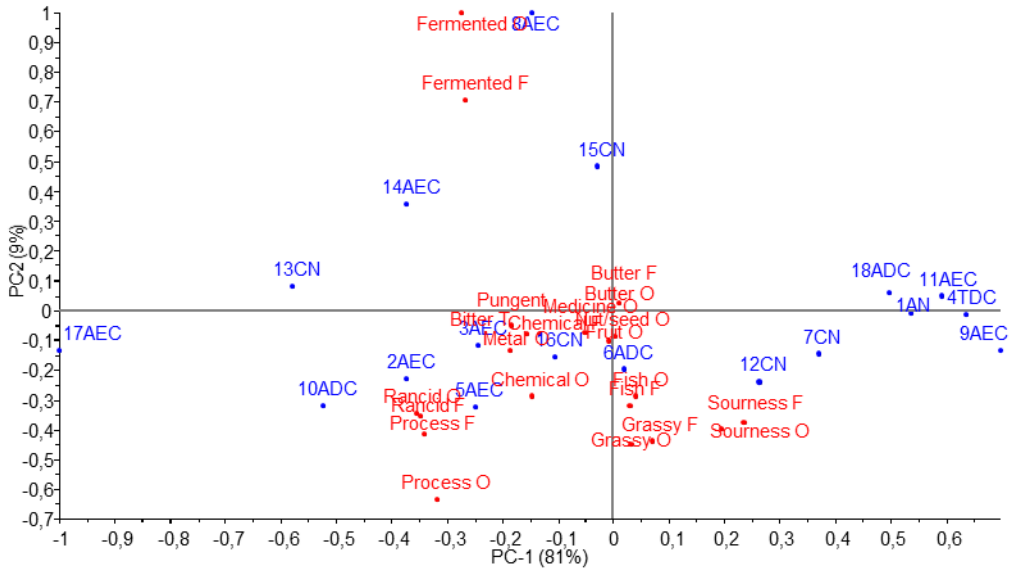


**Figure 2. Principal component analysis (PCA) bi-plot showing similarities and differences between sensory characteristics and fish oils in trial 2.**

We see the same pattern for sensory characteristics as seen in trial 1 where the sensory characteristics, going from one side to the other, is *sourness-grassy-process-rancid*. Apart from a few exceptions between, the oils are significantly differentiated by the same sensory properties in trial 1 and 2. *Fish odour and flavour were not significant* different in trial 2 just as they were in trial 1.

Fish oils 9AEC, 4TDC, 11AEC, 1AN, 18ADC, 7CN and 12CN were mainly described as having sourness and *grassy* odour and flavour whereas fish oil 17AEC was described as having *rancid* odour and flavour and *bitter* taste. Fish oils 13CN, 10ADC, 2AEC, 3AEC, 5AEC, 16CN and 6ADC were described as having chemical and process odour and flavour.

The second principal component is somehow differentiating fish oil 8AEC, 15CN and 14AEC from the other fish oils, and these are described by more fermented odour and flavour.

When considering the use of different raw materials, the process and the different concentrate EPA and DHA in the fish oils, there is no correlations. This is similar to the findings in trial 1. The fish oils are spread all over the plot.

### 3.2.3 TRIAL 3 – STORAGE EXPERIMENT

Eight refined oils were selected from trial 2 and analysed after 7, 14 and 20 days of storage. Table 9 a and b show the results, and among 22 tested sensory characteristics, 17 showed significant differences between the different storage and fish oils (Table 9 a and b).

**Table 9a. Odour values for the fish oils. Mean of x samples shown. Different letters indicate significant difference ($p<0.05$) among the fish oil products using two-way ANOVA and Tukey's multiple comparison test.**

| Sample | Sourness odour | Grassy odour | Fish odour | Butter odour | Metal odour | Fruit odour | Nut/seed odour | Chemical odour | Process odour | Medicine odour | Fermented odour | Rancid odour |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **3AEC-7** | 2.97 $^{abcdef}$ | 1.84 $^{ab}$ | 2.13 $^{ab}$ | 1.29 $^{abc}$ | 3.51 $^{bcdefgh}$ | 1.33 $^{a}$ | 1.16 $^{a}$ | 2.07 $^{a}$ | 1.93 $^{cdefg}$ | 1.25 $^{a}$ | 2.19 $^{bc}$ | 2.66 $^{bcdef}$ |
| **3AEC-14** | 2.59 $^{bcdef}$ | 2.13 $^{ab}$ | 2.45 $^{ab}$ | 1.12 $^{bc}$ | 4.17 $^{abcdef}$ | 1.19 $^{a}$ | 1.06 $^{a}$ | 1.61 $^{a}$ | 2.41 $^{bcdefg}$ | 1.32 $^{a}$ | 1.50 $^{c}$ | 2.97 $^{bcdef}$ |
| **3AEC-20** | 1.88 $^{def}$ | 1.41 $^{ab}$ | 2.17 $^{ab}$ | 1.05 $^{c}$ | 4.89 $^{a}$ | 1.21 $^{a}$ | 1.11 $^{a}$ | 2.53 $^{a}$ | 3.88 $^{abc}$ | 1.33 $^{a}$ | 3.29 $^{abc}$ | 4.26 $^{abc}$ |
| **5AEC-7** | 1.82 $^{ef}$ | 1.57 $^{ab}$ | 1.93 $^{ab}$ | 1.04 $^{c}$ | 4.42 $^{abcd}$ | 1.21 $^{a}$ | 1.17 $^{a}$ | 2.41 $^{a}$ | 4.16 $^{ab}$ | 1.32 $^{a}$ | 2.86 $^{abc}$ | 4.21 $^{abc}$ |
| **5AEC-14** | 2.06 $^{def}$ | 1.61 $^{ab}$ | 2.23 $^{ab}$ | 1.09 $^{c}$ | 4.32 $^{abcde}$ | 1.30 $^{a}$ | 1.17 $^{a}$ | 2.27 $^{a}$ | 2.93 $^{bcdefg}$ | 1.33 $^{a}$ | 2.43 $^{bc}$ | 3.89 $^{abcd}$ |
| **5AEC-20** | 2.13 $^{def}$ | 2.31 $^{ab}$ | 1.99 $^{ab}$ | 1.08 $^{c}$ | 4.52 $^{abc}$ | 1.43 $^{a}$ | 1.33 $^{a}$ | 2.91 $^{a}$ | 3.80 $^{abcd}$ | 1.31 $^{a}$ | 2.57 $^{bc}$ | 4.41 $^{ab}$ |
| **7CN-7** | 4.26 $^{a}$ | 1.95 $^{ab}$ | 2.42 $^{ab}$ | 1.56 $^{abc}$ | 2.75 $^{h}$ | 2.02 $^{a}$ | 1.67 $^{a}$ | 1.66 $^{a}$ | 1.61 $^{efg}$ | 1.09 $^{a}$ | 1.24 $^{c}$ | 1.58 $^{ef}$ |
| **7CN-14** | 4.36 $^{a}$ | 2.63 $^{a}$ | 2.29 $^{ab}$ | 1.41 $^{abc}$ | 2.78 $^{h}$ | 1.52 $^{a}$ | 1.54 $^{a}$ | 1.49 $^{a}$ | 1.22 $^{g}$ | 1.19 $^{a}$ | 1.19 $^{c}$ | 1.46 $^{f}$ |
| **7CN-20** | 3.49 $^{abcd}$ | 2.14 $^{ab}$ | 2.11 $^{ab}$ | 1.57 $^{abc}$ | 2.89 $^{gh}$ | 1.69 $^{a}$ | 1.53 $^{a}$ | 2.00 $^{a}$ | 1.82 $^{cdefg}$ | 1.41 $^{a}$ | 1.53 $^{c}$ | 1.69 $^{ef}$ |
| **8AEC-7** | 1.97 $^{def}$ | 1.43 $^{ab}$ | 2.42 $^{ab}$ | 1.02 $^{c}$ | 3.91 $^{abcdefgh}$ | 1.33 $^{a}$ | 1.27 $^{a}$ | 1.99 $^{a}$ | 2.37 $^{bcdefg}$ | 1.34 $^{a}$ | 2.83 $^{abc}$ | 3.49 $^{abcde}$ |
| **8AEC-14** | 1.94 $^{def}$ | 1.45 $^{ab}$ | 2.13 $^{ab}$ | 1.06 $^{c}$ | 4.42 $^{abcd}$ | 1.26 $^{a}$ | 1.32 $^{a}$ | 2.14 $^{a}$ | 3.66 $^{abcde}$ | 1.41 $^{a}$ | 2.64 $^{abc}$ | 4.43 $^{ab}$ |
| **8AEC-20** | 1.73 $^{f}$ | 1.31 $^{b}$ | 2.03 $^{ab}$ | 1.06 $^{c}$ | 4.61 $^{ab}$ | 1.32 $^{a}$ | 1.45 $^{a}$ | 2.46 $^{a}$ | 3.66 $^{abcde}$ | 1.49 $^{a}$ | 2.84 $^{abc}$ | 4.64 $^{ab}$ |
| **11AEC-7** | 4.03 $^{ab}$ | 2.63 $^{a}$ | 2.33 $^{ab}$ | 1.37 $^{abc}$ | 3.19 $^{defgh}$ | 2.19 $^{a}$ | 1.40 $^{a}$ | 1.99 $^{a}$ | 1.26 $^{g}$ | 1.62 $^{a}$ | 1.21 $^{c}$ | 1.44 $^{f}$ |
| **11AEC-14** | 2.97 $^{abcdef}$ | 2.04 $^{ab}$ | 2.07 $^{ab}$ | 1.21 $^{bc}$ | 3.28 $^{cdefgh}$ | 1.28 $^{a}$ | 1.22 $^{a}$ | 1.63 $^{a}$ | 2.46 $^{bcdefg}$ | 1.37 $^{a}$ | 1.99 $^{c}$ | 2.87 $^{bcdef}$ |
| **11AEC-20** | 2.50 $^{bcdef}$ | 1.83 $^{ab}$ | 2.13 $^{ab}$ | 1.06 $^{c}$ | 3.90 $^{abcdefgh}$ | 1.47 $^{a}$ | 1.24 $^{a}$ | 2.40 $^{a}$ | 2.88 $^{bcdefg}$ | 1.41 $^{a}$ | 2.64 $^{abc}$ | 3.52 $^{abcde}$ |
| **13CN-7** | 1.57 $^{f}$ | 1.57 $^{ab}$ | 1.26 $^{b}$ | 1.13 $^{bc}$ | 4.17 $^{abcdef}$ | 1.18 $^{a}$ | 1.29 $^{a}$ | 2.00 $^{a}$ | 4.19 $^{ab}$ | 1.47 $^{a}$ | 4.78 $^{a}$ | 4.16 $^{abc}$ |
| **13CN-14** | 2.26 $^{cdef}$ | 1.96 $^{ab}$ | 1.91 $^{ab}$ | 1.15 $^{bc}$ | 4.26 $^{abcde}$ | 1.12 $^{a}$ | 1.38 $^{a}$ | 1.69 $^{a}$ | 3.48 $^{abcdef}$ | 1.45 $^{a}$ | 2.71 $^{abc}$ | 4.51 $^{ab}$ |
| **13CN-20** | 1.48 $^{f}$ | 1.28 $^{b}$ | 1.66 $^{ab}$ | 1.01 $^{c}$ | 4.88 $^{a}$ | 1.14 $^{a}$ | 1.25 $^{a}$ | 2.62 $^{a}$ | 5.03 $^{a}$ | 1.67 $^{a}$ | 4.30 $^{ab}$ | 5.22 $^{a}$ |
| **15CN-7** | 2.48 $^{bcdef}$ | 1.32 $^{b}$ | 1.49 $^{ab}$ | 1.95 $^{abc}$ | 3.61 $^{bcdefgh}$ | 1.96 $^{a}$ | 1.01 $^{a}$ | 2.67 $^{a}$ | 1.88 $^{cdefg}$ | 1.28 $^{a}$ | 3.09 $^{abc}$ | 2.96 $^{bcdef}$ |
| **15CN-14** | 3.39 $^{abcde}$ | 1.92 $^{ab}$ | 2.53 $^{a}$ | 2.22 $^{ab}$ | 3.07 $^{efgh}$ | 2.33 $^{a}$ | 1.36 $^{a}$ | 2.07 $^{a}$ | 1.71 $^{efg}$ | 1.07 $^{a}$ | 2.11 $^{c}$ | 2.35 $^{cdef}$ |
| **15CN-20** | 3.79 $^{abc}$ | 1.68 $^{ab}$ | 2.58 $^{a}$ | 2.32 $^{a}$ | 2.97 $^{fgh}$ | 2.12 $^{a}$ | 1.64 $^{a}$ | 1.63 $^{a}$ | 1.41 $^{fg}$ | 1.19 $^{a}$ | 1.94 $^{c}$ | 1.88 $^{def}$ |
| **16CN-7** | 3.79 $^{abc}$ | 2.06 $^{ab}$ | 2.36 $^{ab}$ | 1.44 $^{abc}$ | 2.82 $^{h}$ | 2.02 $^{a}$ | 1.41 $^{a}$ | 1.96 $^{a}$ | 1.74 $^{defg}$ | 1.41 $^{a}$ | 1.64 $^{c}$ | 2.04 $^{def}$ |
| **16CN-14** | 2.24 $^{cdef}$ | 1.68 $^{ab}$ | 2.39 $^{ab}$ | 1.14 $^{bc}$ | 4.11 $^{abcdefg}$ | 1.38 $^{a}$ | 1.11 $^{a}$ | 1.98 $^{a}$ | 2.89 $^{bcdefg}$ | 1.69 $^{a}$ | 1.97 $^{c}$ | 3.37 $^{abcdef}$ |
| **16CN-20** | 2.09 $^{def}$ | 1.74 $^{ab}$ | 2.02 $^{ab}$ | 1.12 $^{bc}$ | 4.14 $^{abcdefg}$ | 1.16 $^{a}$ | 1.03 $^{a}$ | 1.82 $^{a}$ | 2.83 $^{bcdefg}$ | 1.33 $^{a}$ | 1.98 $^{c}$ | 3.24 $^{abcdef}$ |
| **p-value** | <0.001 | <0.001 | 0.022 | <0.001 | <0.001 | 0.001 | 0.139 | 0.164 | <0.001 | 0.711 | <0.001 | <0.001 |

**Table 9b. Flavour, taste and mouthfeel values for the fish oils. Mean of x samples shown. Different letters indicate significant difference (p<0.05) among the fish oil products using two-way ANOVA and Tukey's multiple comparison test.**

| Sample | Sourness flavour | Bitter taste | Grassy flavour | Fish flavour | Butter flavour | Chemical flavour | Process flavour | Fermented flavour | Rancid flavour | Pungent |
|---|---|---|---|---|---|---|---|---|---|---|
| 3AEC-7 | 2.91 $^{abcdefg}$ | 3.82 $^{abcd}$ | 1.96 $^{abcdef}$ | 2.19 $^{ab}$ | 1.16 $^{a}$ | 2.32 $^{a}$ | 2.66 $^{bcde}$ | 2.26 $^{cd}$ | 2.10 $^{cdef}$ | 3.67 $^{abc}$ |
| 3AEC-14 | 2.72 $^{bcdefg}$ | 4.01 $^{abcd}$ | 1.83 $^{bcdef}$ | 2.79 $^{a}$ | 1.10 $^{a}$ | 1.58 $^{a}$ | 2.36 $^{cde}$ | 1.40 $^{cd}$ | 2.87 $^{abcdef}$ | 3.64 $^{abc}$ |
| 3AEC-20 | 1.70 $^{fg}$ | 4.43 $^{abc}$ | 1.33 $^{def}$ | 2.23 $^{ab}$ | 1.06 $^{a}$ | 2.44 $^{a}$ | 4.18 $^{abc}$ | 2.97 $^{cd}$ | 4.11 $^{abc}$ | 3.79 $^{abc}$ |
| 5AEC-7 | 1.97 $^{efg}$ | 4.60 $^{ab}$ | 1.66 $^{bcdef}$ | 1.82 $^{ab}$ | 1.10 $^{a}$ | 2.71 $^{a}$ | 4.22 $^{abc}$ | 2.57 $^{cd}$ | 4.50 $^{a}$ | 4.01 $^{abc}$ |
| 5AEC-14 | 2.34 $^{defg}$ | 3.79 $^{abcd}$ | 1.73 $^{bcdef}$ | 2.38 $^{ab}$ | 1.27 $^{a}$ | 2.41 $^{a}$ | 2.94 $^{abcde}$ | 2.28 $^{cd}$ | 3.64 $^{abcde}$ | 3.78 $^{abc}$ |
| 5AEC-20 | 1.57 $^{fg}$ | 4.21 $^{abcd}$ | 1.51 $^{cdef}$ | 1.95 $^{ab}$ | 1.12 $^{a}$ | 3.31 $^{a}$ | 4.04 $^{abcd}$ | 2.91 $^{cd}$ | 4.49 $^{a}$ | 4.37 $^{abc}$ |
| 7CN-7 | 4.58 $^{a}$ | 2.97 $^{d}$ | 3.17 $^{a}$ | 2.64 $^{a}$ | 1.23 $^{a}$ | 1.99 $^{a}$ | 1.41 $^{e}$ | 1.13 $^{d}$ | 1.49 $^{f}$ | 3.01 $^{bc}$ |
| 7CN-14 | 4.26 $^{abc}$ | 3.23 $^{cd}$ | 2.76 $^{ab}$ | 2.77 $^{a}$ | 1.52 $^{a}$ | 1.76 $^{a}$ | 1.51 $^{e}$ | 1.43 $^{cd}$ | 1.67 $^{ef}$ | 2.95 $^{bc}$ |
| 7CN-20 | 3.73 $^{abcde}$ | 3.24 $^{cd}$ | 2.49 $^{abcd}$ | 2.56 $^{ab}$ | 1.33 $^{a}$ | 1.77 $^{a}$ | 1.74 $^{de}$ | 1.27 $^{cd}$ | 1.87 $^{def}$ | 2.94 $^{bc}$ |
| 8AEC-7 | 2.52 $^{bcdefg}$ | 4.14 $^{abcd}$ | 1.81 $^{bcdef}$ | 2.46 $^{ab}$ | 1.36 $^{a}$ | 2.20 $^{a}$ | 2.56 $^{bcde}$ | 2.95 $^{cd}$ | 3.25 $^{abcdef}$ | 3.44 $^{abc}$ |
| 8AEC-14 | 1.93 $^{efg}$ | 4.43 $^{abc}$ | 1.69 $^{bcdef}$ | 2.25 $^{ab}$ | 1.09 $^{a}$ | 1.74 $^{a}$ | 3.58 $^{abcde}$ | 2.18 $^{cd}$ | 4.26 $^{ab}$ | 4.28 $^{abc}$ |
| 8AEC-20 | 1.91 $^{efg}$ | 4.07 $^{abcd}$ | 1.40 $^{def}$ | 1.93 $^{ab}$ | 1.08 $^{a}$ | 1.74 $^{a}$ | 3.96 $^{abcd}$ | 3.01 $^{bcd}$ | 4.41 $^{ab}$ | 3.58 $^{abc}$ |
| 11AEC-7 | 4.34 $^{ab}$ | 3.40 $^{bcd}$ | 2.66 $^{abc}$ | 2.79 $^{a}$ | 1.29 $^{a}$ | 1.74 $^{a}$ | 1.48 $^{e}$ | 1.28 $^{cd}$ | 1.38 $^{f}$ | 3.11 $^{abc}$ |
| 11AEC-14 | 2.62 $^{bcdefg}$ | 3.92 $^{abcd}$ | 2.06 $^{abcdef}$ | 2.01 $^{ab}$ | 1.16 $^{a}$ | 2.44 $^{a}$ | 2.89 $^{bcde}$ | 1.83 $^{cd}$ | 3.14 $^{abcdef}$ | 4.01 $^{abc}$ |
| 11AEC-20 | 2.29 $^{defg}$ | 4.30 $^{abcd}$ | 1.69 $^{bcdef}$ | 2.28 $^{ab}$ | 1.16 $^{a}$ | 2.23 $^{a}$ | 3.04 $^{abcde}$ | 2.39 $^{cd}$ | 3.74 $^{abcd}$ | 4.41 $^{ab}$ |
| 13CN-7 | 1.17 $^{fg}$ | 4.74 $^{a}$ | 1.15 $^{ef}$ | 1.04 $^{b}$ | 1.03 $^{a}$ | 2.34 $^{a}$ | 4.68 $^{ab}$ | 5.67 $^{a}$ | 4.48 $^{a}$ | 4.63 $^{a}$ |
| 13CN-14 | 1.77 $^{fg}$ | 4.32 $^{abc}$ | 1.33 $^{def}$ | 1.96 $^{ab}$ | 1.12 $^{a}$ | 1.71 $^{a}$ | 4.11 $^{abc}$ | 3.26 $^{bcd}$ | 4.29 $^{ab}$ | 3.88 $^{abc}$ |
| 13CN-20 | 1.08 $^{g}$ | 4.77 $^{a}$ | 1.04 $^{f}$ | 1.43 $^{ab}$ | 1.02 $^{a}$ | 1.97 $^{a}$ | 5.23 $^{a}$ | 5.32 $^{ab}$ | 4.72 $^{a}$ | 4.67 $^{a}$ |
| 15CN-7 | 2.46 $^{cdefg}$ | 3.84 $^{abcd}$ | 1.72 $^{bcdef}$ | 1.61 $^{ab}$ | 1.38 $^{a}$ | 3.12 $^{a}$ | 2.23 $^{cde}$ | 3.48 $^{abc}$ | 2.80 $^{abcdef}$ | 3.79 $^{abc}$ |
| 15CN-14 | 2.67 $^{bcdefg}$ | 3.26 $^{cd}$ | 1.66 $^{bcdef}$ | 2.35 $^{ab}$ | 1.45 $^{a}$ | 2.76 $^{a}$ | 2.21 $^{cde}$ | 2.43 $^{cd}$ | 2.40 $^{bcdef}$ | 3.49 $^{abc}$ |
| 15CN-20 | 2.95 $^{abcdef}$ | 3.31 $^{bcd}$ | 1.87 $^{bcdef}$ | 2.13 $^{ab}$ | 1.49 $^{a}$ | 2.24 $^{a}$ | 2.00 $^{cde}$ | 2.53 $^{cd}$ | 2.69 $^{abcdef}$ | 3.09 $^{abc}$ |
| 16CN-7 | 3.82 $^{abcd}$ | 3.39 $^{bcd}$ | 2.34 $^{abcde}$ | 2.54 $^{ab}$ | 1.18 $^{a}$ | 1.83 $^{a}$ | 2.25 $^{cde}$ | 1.58 $^{cd}$ | 1.82 $^{def}$ | 2.79 $^{c}$ |
| 16CN-14 | 2.75 $^{abcdefg}$ | 4.01 $^{abcd}$ | 1.72 $^{bcdef}$ | 2.22 $^{ab}$ | 1.33 $^{a}$ | 1.82 $^{a}$ | 2.68 $^{bcde}$ | 1.87 $^{cd}$ | 3.09 $^{abcdef}$ | 3.49 $^{abc}$ |
| 16CN-20 | 1.91 $^{efg}$ | 3.69 $^{abcd}$ | 1.64 $^{bcdef}$ | 2.16 $^{ab}$ | 1.12 $^{a}$ | 1.63 $^{a}$ | 2.87 $^{bcde}$ | 1.91 $^{cd}$ | 3.40 $^{abcdef}$ | 3.78 $^{abc}$ |
| p-verdi | <0.001 | <0.001 | <0.001 | 0.005 | 0.403 | 0.059 | <0.001 | <0.001 | <0.001 | <0.001 |

Principal component analysis (PCA) was used to find similarities and differences between the sensory characteristics, storage times and the fish oils (Figure 3 and 4). The first and second principal components (PCs) explained 81 % and 8 % of the plot, respectively, which means the first PCs dominate the interpretation of the plots moving in the direction from *rancid*/*process* to *sourness*.

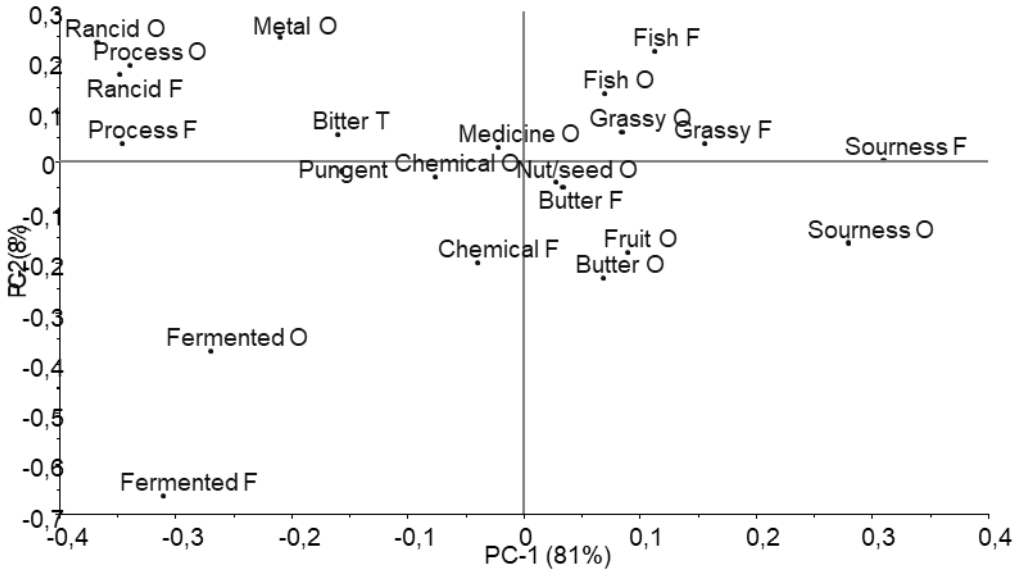


**Figure 3. Principal component analysis (PCA) loading-plot showing similarities and differences between sensory characteristics, storage times and fish oils in Trial 3.**

Again, we see the same pattern with regard to how the sensory characteristics distinguish the different samples (Figure 3 and Figure 4). The different fish oils have different resistance to oxidation during storage, as can be seen in Figure 4. During storage fish oils 11AEC, 16CN and 3AEC were evaluated with higher intensity of rancid odour and flavour and bitter taste. Fish oils 7CN, 15CN, 5AEC, 8AEC and 13CN have different starting points, but are stable against oxidation during storage.

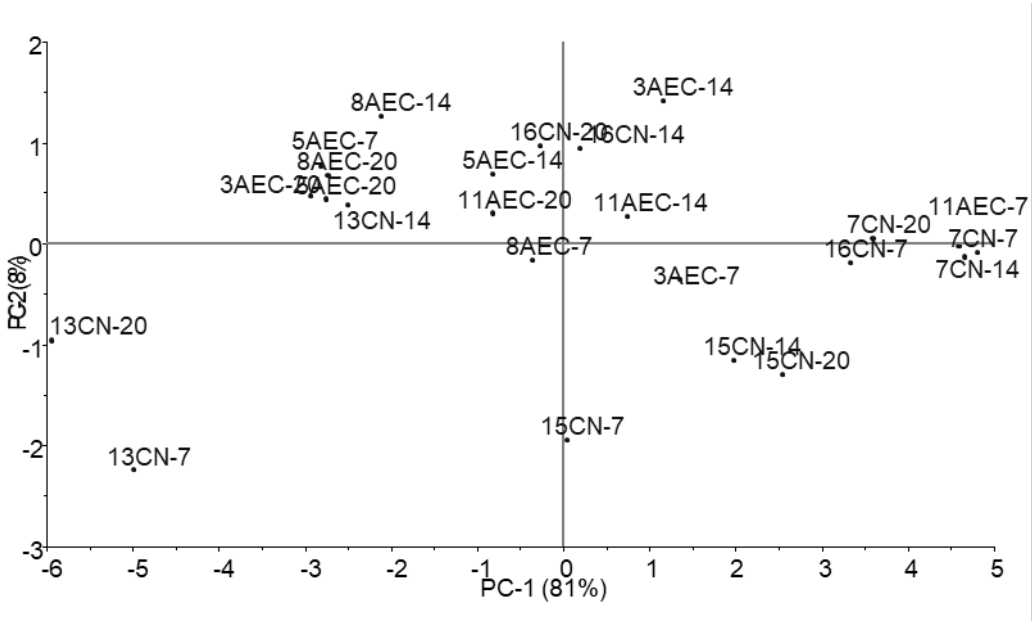


**Figure 4. Principal component analysis (PCA) score-plot showing similarities and differences between sensory characteristics, storage times and fish oils in Trial 3.**

When considering the use of different raw materials and the different concentrates, no significant correlations are found. The different fish oils are spread all over the plot and, and as mentioned above, they have different starting points. This is in accordance with the results from Trials 1 and 2.

## 3.3 THE OXIDATION PROPERTIES PEROXIDE VALUE, ANISIDINE VALUE AND FREE FATTY ACID

### 3.3.1 TRIAL 1

Table 10 shows that the amounts of free fatty acids in the samples in Trial 1 are low. Only sample 8CC_17 exceeds the suggested limit of 0.25 % in GOED guidance documents. Peroxide values in this trial are low, and all are below the GOED maximum value of 5 meq/kg oil. The cod liver oils have, with one exception, the lowest peroxide values compared to the other oils. The anisidine values exhibit larger variations between the samples, but none of the samples exceed the GOED maximum value of 20.

**Table 10. Free fatty acid (FFA), peroxide (PV), anisidine (AnV), absorbance and colour for the different fish oils collected in 2017. Results shown as averages of duplicates.**

| Sample | FFA (%) | PV (meq peroxides/kg oil) | AnV | Absorbance | Colour (Gardner) |
|---|---|---|---|---|---|
| 1CN_17 | 0.13 | 4.1 | 8.3 | 0.6 | 3.3 |
| 7CN_17 | 0.06 | 0.7 | 10.3 | 0.7 | 3.5 |
| 15CN_17 | 0.13 | 0.0 | 2.5 | 0.8 | 3.7 |
| 16CN_17 | 0.07 | 0.7 | 7.0 | 0.7 | 2.0 |
| 18CN_17 | 0.04 | 0.6 | 1.4 | 0.7 | 1.7 |
| 19CN_17 | 0.04 | 0.6 | 1.4 | 0.7 | 2.0 |
| 8CC_17 | 0.29 | 1.3 | 14.0 | 0.9 | 4.3 |
| 20AN_17 | 0.05 | 1.4 | 8.2 | 0.6 | 4.3 |
| 3AEC_17 | 0.10 | 2.7 | 11.4 | 0.8 | 3.5 |
| 4AEC_17 | 0.04 | 1.3 | 3.2 | 0.6 | 3.5 |
| 17AEC _17 | 0.07 | 1.4 | 6.3 | 1.0 | 2.0 |
| 21AEC_17 | 0.08 | 1.2 | 4.1 | 0.7 | 1.5 |
| 22AEC_17 | 0.05 | 2.1 | 4.4 | 1.0 | 2.0 |
| 23AEC_17 | 0.06 | 1.8 | 3.4 | 1.2 | 2.5 |
| 5ADC_17 | 0.06 | 1.9 | 2.9 | 0.8 | 3.5 |
| 6ADC_17 | 0.08 | 3.6 | 5.4 | 1.0 | 4.3 |
| 12ADC_17 | 0.04 | 0.3 | 3.8 | 1.0 | 3.5 |

### 3.3.2 TRIAL 2

Table 11 presents the results from Trial 2. The results demonstrate that the oil samples are of good quality compared to the maximum values for the quality parameters given by GOED. The peroxide values in Trial 2 tends to be lower than in trial 1.

**Table 11. Free fatty acid (FFA), peroxide (PV), anisidine (AnV), absorbance and colour for the different fish oils collected in 2018. Results shown as averages of duplicates.**

| Sample | FFA (%) | PV (meq peroxyd/kg oil) | AnV | Absorbance | Colour (Gardner) |
|---|---|---|---|---|---|
| 7CN_18 | 0.05 | 0.5 | 3.7 | 0.7 | 2.0 |
| 12CN_18 | 0.05 | 0.9 | 4.6 | 0.7 | 1.5 |
| 13CN_18 | 0.17 | 1.4 | 14.9 | 0.6 | 3.5 |
| 15CN_18 | 0.07 | 0.3 | 3.6 | 0.7 | 3.5 |
| 16CN_18 | 0.05 | 2.4 | 2.8 | 0.7 | 1.5 |
| 1AN_18 | 0.07 | 0.5 | 7.3 | 0.7 | 4.0 |
| 2AEC_18 | 0.11 | 0.4 | 4.5 | 0.8 | 1.0 |
| 3AEC_18 | 0.11 | 0.8 | 5.0 | 0.8 | 1.0 |
| 5AEC_18 | 0.08 | 2.8 | 5.1 | 1.1 | 1.5 |
| 8AEC_18 | 0.04 | 0.8 | 1.4 | 0.6 | 1.5 |
| 9AEC_18 | 0.12 | 0.2 | 1.9 | 0.7 | 1.5 |
| 11AEC_18 | 0.20 | 0.2 | 2.2 | 0.8 | 3.5 |
| 14AEC_18 | 0.11 | 1.3 | 2.8 | 0.7 | 3.5 |
| 17AEC_18 | 0.16 | 3.0 | 17.3 | 0.8 | 5.0 |
| 6ADC_18 | 0.10 | 0.9 | 4.4 | 0.9 | 4.0 |
| 10ADC_18 | 0.10 | 3.5 | 4.0 | 1.2 | 1.5 |
| 18ADC_18 | 0.11 | 0.4 | 13.9 | 1.5 | 4.0 |
| 4TDC_18 | 0.21 | 0.2 | 4.3 | 1.0 | 4.0 |

### 3.3.3 TRIAL 3 – STORAGE EXPERIMENT

The storage study in Trial 3, presented in Table 12, demonstrates the development of oxidation in the different oils. All oils have increasing peroxide and anisidine values during storage (Figure 5 and 6). The GOED maximum value for the peroxide value is exceeded within one week of storage for most of the samples. The increase in anisidine value is slower and do not exceed the GOED maximum value during 3 weeks of storage. The changes in free fatty acids are not uniform among the samples.

**Table 12. Free fatty acid (FFA), peroxide (PV), anisidine (AnV), absorbance and colour for the different fish oils during storage. Results shown as averages of duplicates.**

| Sample | FFA (%) | PV (meq peroxyd/kg oil) | AnV | Absorbance | Colour (Gardner) |
|---|---|---|---|---|---|
| 7CN_18_1 | 0.06 | 1.8 | 7.1 | 0.9 | 3.5 |
| 7CN_18_2 | 0.06 | 2.7 | 7.5 | 0.9 | 3.5 |
| 7CN_18_3 | 0.05 | 5.9 | 8.2 | 0.9 | 3.5 |
| 13CN_18_1 | 0.20 | 4.6 | 10,0 | 0.7 | 3.5 |
| 13CN_18_2 | 0.14 | 8.3 | 15.8 | 0.8 | 3.5 |
| 13CN_18_3 | 0.14 | 14.3 | 17.8 | 0.7 | 3.5 |
| 15CN_18_1 | 0.11 | 1.9 | 2.1 | 0.8 | 3.5 |
| 15CN_18_2 | 0.05 | 3.6 | 3.9 | 0.8 | 3.5 |
| 15CN_18_3 | 0.05 | 5.1 | 4.3 | 0.8 | 3.5 |
| 16CN_18_1 | 0.12 | 8.4 | 4.6 | 0.8 | 2.0 |
| 16CN_18_2 | 0.05 | 12.7 | 6.7 | 0.9 | 2.5 |
| 16CN_18_3 | 0.05 | 16.8 | 8.2 | 0.9 | 2.5 |
| 3AEC_18_1 | 0.09 | 9.1 | 5.1 | 0.9 | 1.5 |
| 3AEC_18_2 | 0.08 | 14.0 | 6.4 | 1.0 | 1.5 |
| 3AEC_18_3 | 0.08 | 20.4 | 6.7 | 1.0 | 1.5 |
| 5AEC_18_1 | 0.06 | 11.4 | 4.8 | 1.2 | 1.5 |
| 5AEC_18_2 | 0.05 | 18.8 | 6.2 | 1.3 | 1.5 |
| 5AEC_18_3 | 0.05 | 30.0 | 8.1 | 1.4 | 2.0 |
| 8AEC_18_1 | 0.06 | 9.6 | 2.2 | 0.8 | 1.0 |
| 8AEC_18_2 | 0.04 | 16.1 | 2.9 | 0.9 | 1.5 |
| 8AEC_18_3 | 0.04 | 24.2 | 5.5 | 1.1 | 1.5 |
| 11AEC_18_1 | 0.25 | 2.5 | 2.3 | 0.9 | 2.0 |
| 11AEC_18_2 | 0.32 | 6.5 | 2.7 | 1.0 | 2.0 |
| 11AEC_18_3 | 0.53 | 15.4 | 4.5 | 1.0 | 2.5 |

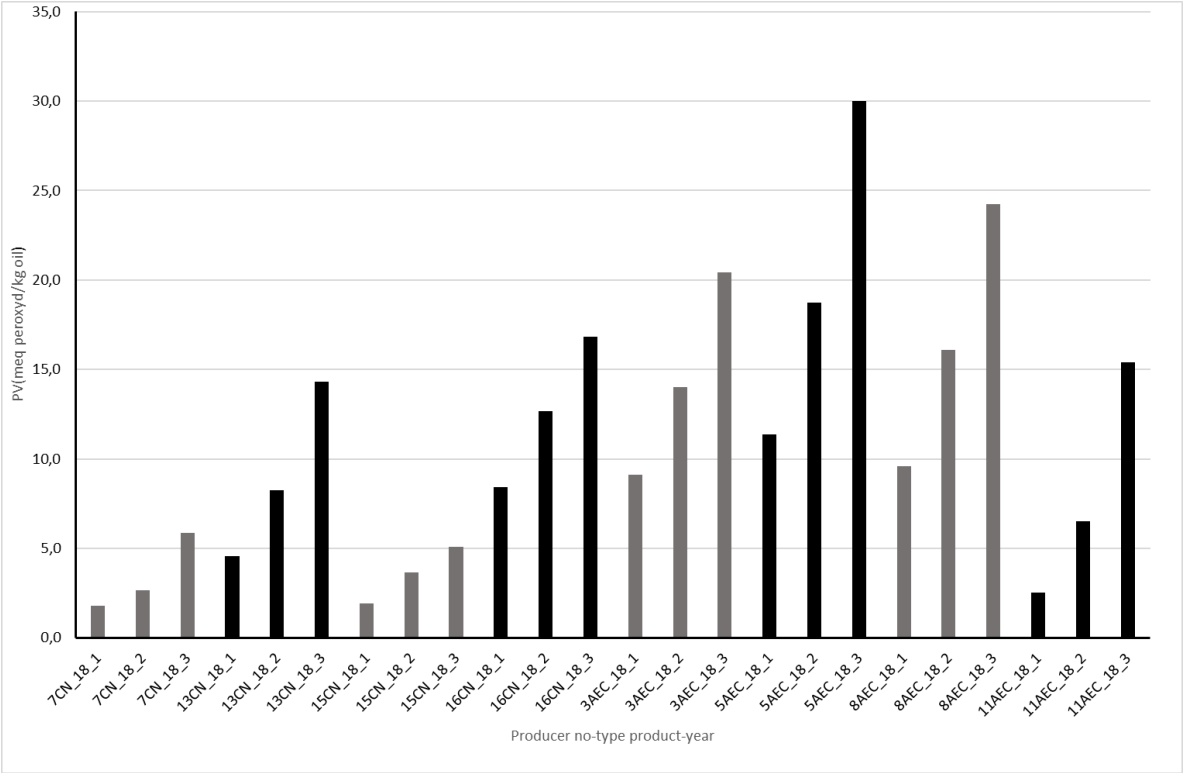


**Figure 5. Peroxide (PV) value through three weeks of storage. N=8x3**

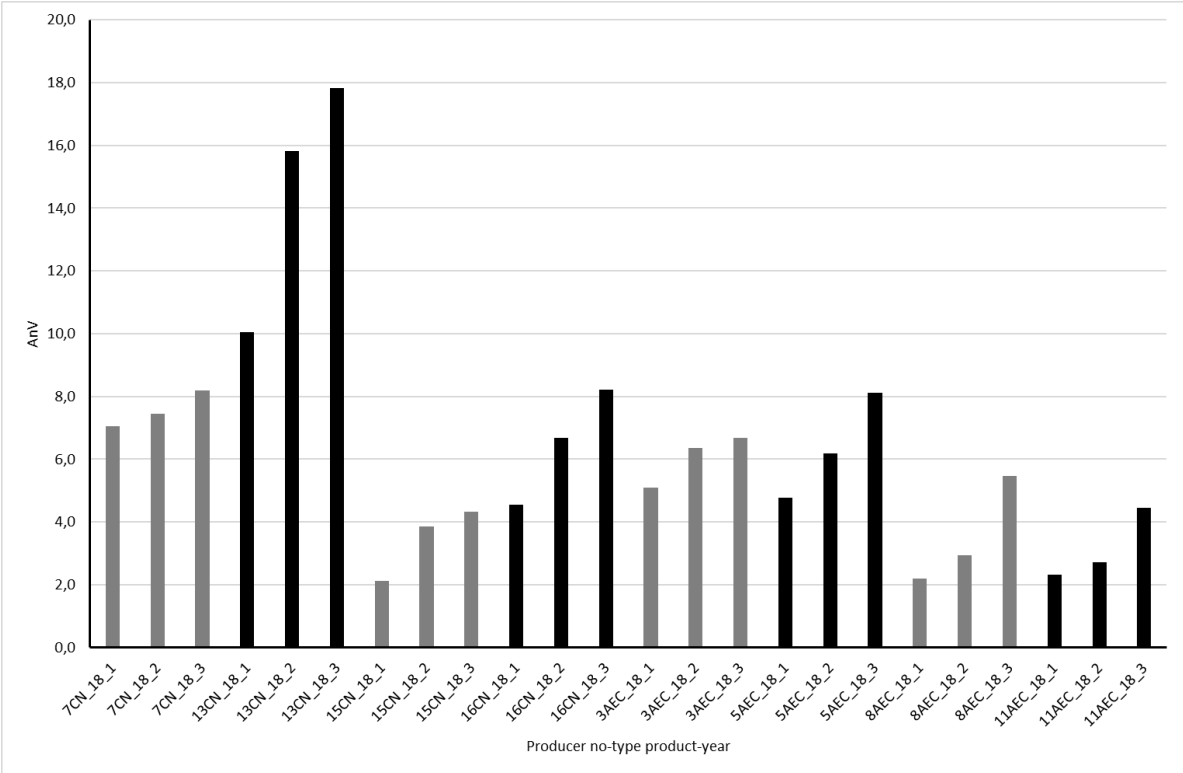


**Figure 6. Anisidine (AnV) value through three weeks of storage. N=8x3**

### 3.4 ABSORBANCE AND COLOUR

Tables 10 and 11 show small variations in the absorbance values of the oils in Trial 1 and Trial 2. Few samples are below the GOED guidance limit of 0.7. Table 12 shows that storage increases the absorbance values for some of the samples. The colour values vary among the samples from 1 to 5 units, as shown in Tables 10, 11 and 12. There is no correlation between colour and the other oxidation parameters.

### 3.5 ASSOCIATION BETWEEN SENSORY PROPERTIES AND THE OXIDATION PROPERTIES PEROXIDE, ANISIDINE, FREE FATTY ACID, COLOUR AND ABSORBANCE

In trial 1 multivariate regression (PLSR) between the chemical oxidation products and the sensory scores of all oils together gave a significant ($p<0.05$) correlation for peroxide value and free fatty acid ($r=0.66$), but no correlation for anisidine values. Looking at the different parameters separately there was no correlation between the sensory characteristics and the anisidine value, while there was a negative correlation between the peroxide value and *sourness* ($r=-0,64$) and a positive correlation between the peroxide value and metal, process and rancid ($r= 0,65-0,73$) odour and flavour. For colour and absorbance there was no correlation with the sensory data or the chemical oxidation products.

In trial 2 multivariate regression (PLSR) between the chemical oxidation products and sensory scores of all oils together gave a significant ($p<0.05$) correlation for anisidine and peroxide level ($r=0.78–0.86$), but no correlation for free fatty acids. Figure 7 illustrates one of the correlations between a sensory characteristic and the anisidine value. Three samples were separated from the rest and probably responsible for the high correlation. Looking at the different parameters separately there was no correlation between the sensory characteristics and the anisidine value, while there was a positive correlation between the peroxide value and chemistry, process and rancid ($r= 0.66–0.78$) odour and flavour. There was also a weak correlation between the colour and the anisidine value ($r=0.55$).

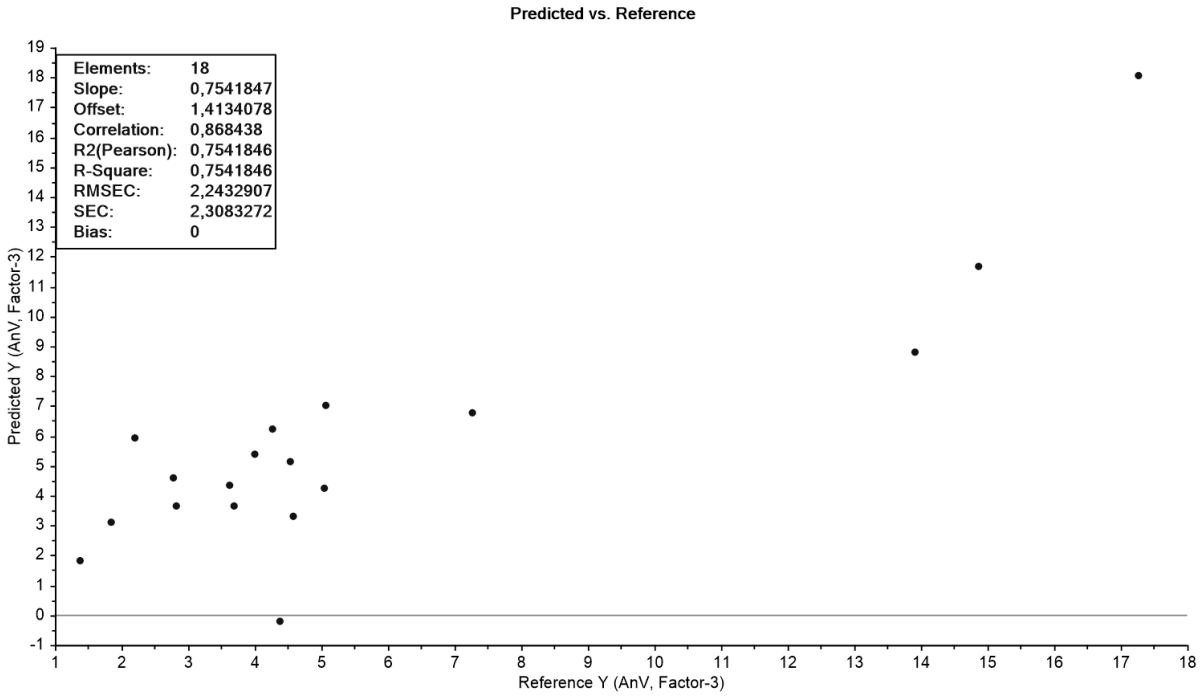


**Figure 7. The predicted vs. reference plot between all sensory characteristic and the anisidine value in trial 2. N= 18.**

In trial 3 multivariate regression (PLSR) between the chemical oxidation products and sensory scores for all oils together gave a significant ($p<0.0005$) correlation for peroxide level ($r=0.75$), but no correlation for anisidine and free fatty acids. Looking at the different parameters separately there was no correlation between the sensory characteristics and the anisidine value, while there was a positive correlation between the peroxide value and metallic and rancid ($r= 0.62–0.68$) odour and flavour.

Multivariate regression (PLSR) between the colour and absorbance and sensory scores of all oils together gave a significant ($p<0.0005$) correlation for colour ($r=0.69$) and absorbance (0.56). There was also a positive correlation between absorbance and peroxide value ($r=0.65$).

## 3.6 VOLATILES

### 3.6.1 TRIAL 1

18 refined oils and 10 oils that had not yet been deodorized, were analysed. Oil samples consisted of up to about 100 volatile compounds, among which about 80 % could be identified. They were dominated by volatile secondary lipid oxidation products from unsaturated fatty acids: propanal, 1-penten-3-ol, 2,4-octadiene, 1-penten-3-one, 2-pentenal, 3,5-octadiene, 2-pentene, 2-propenal, 2-butenal, 2-ethyl furan, 2,4-heptadienal and acetic acid. Prior to deodorization the oils also contained typical volatile tertiary end products of lipid oxidation (C2–C6 acids) and lipid thermal breakdown products from the refining process.

A typical gas chromatogram from the headspace GC/MS analysis of an anchoveta oil before deodorization is shown in Figure 8. As would be expected, oils prior to deodorization showed in general more compounds and higher levels of oxidation compared to the respective deodorized oils, as shown in Figure 9. Oils of different raw materials, and natural oils versus concentrate oils, also showed differences in their volatile compound profiles, reflecting differences in fatty acid composition, which determines the formation of volatile secondary lipid oxidation products. This fact could also explain the poor correlation between the levels of volatile compounds and AnV values when all the oils together were compared with AnV results.

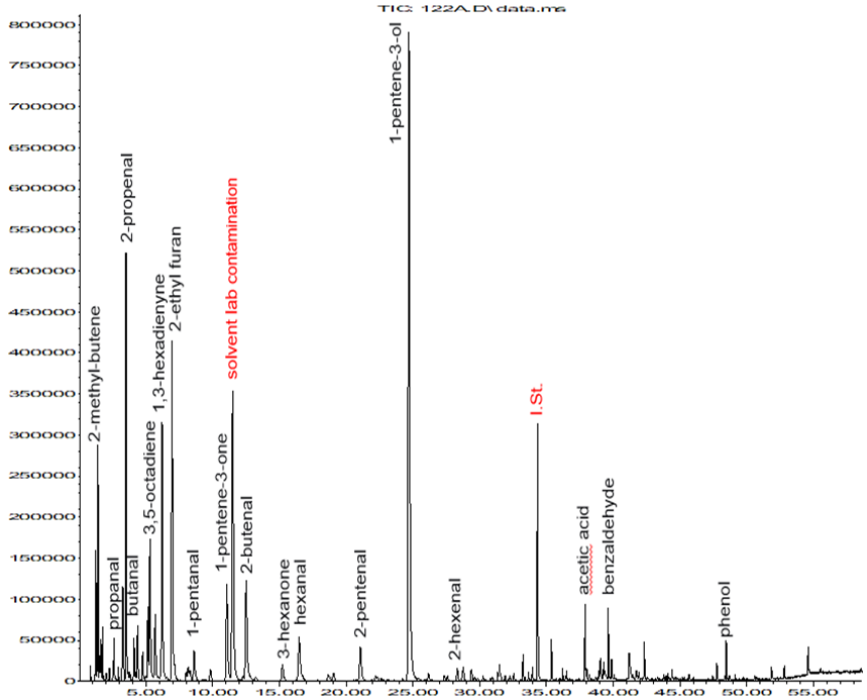


**Figure 8. Gas chromatogram for an anchoveta oil before deodorization.**

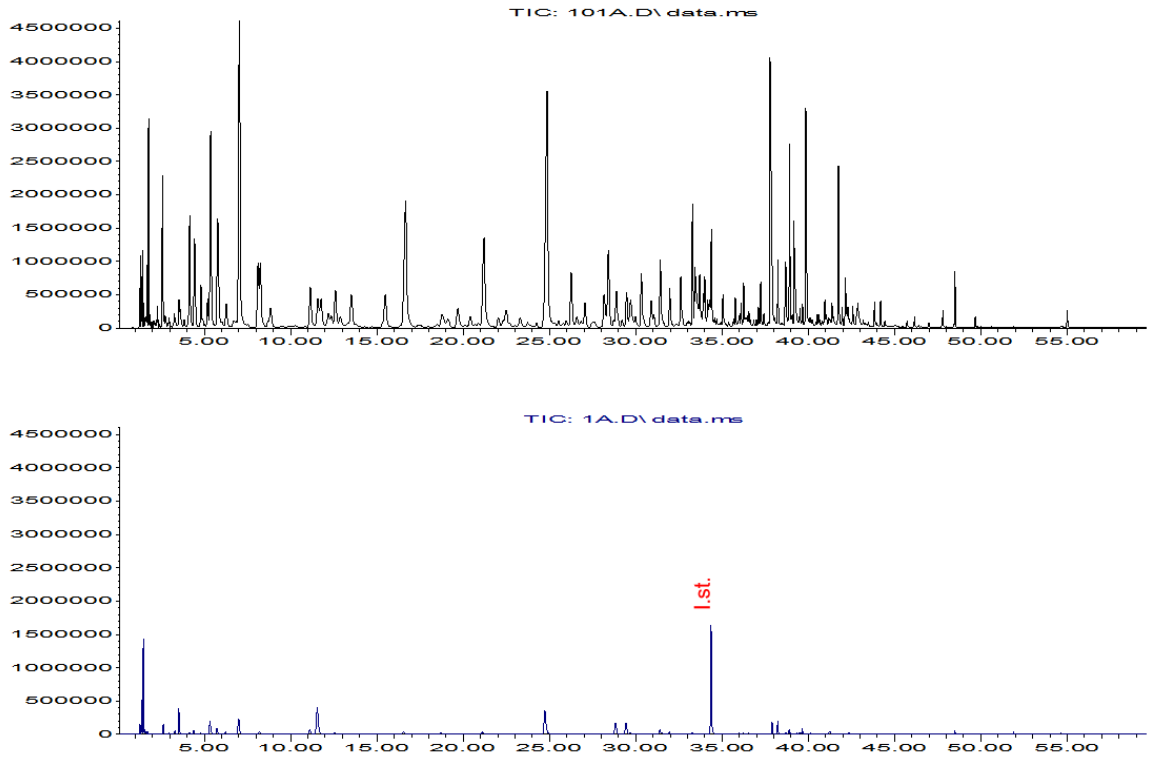


**Figure 9. Gas chromatogram for a natural cod liver oil before and after deodorization.**

### 3.6.2 TRIAL 2

18 new refined oils were analysed. Again, the secondary lipid oxidation products found in Trial 1 dominated the volatile compound profiles of the oils: 1-penten-3-ol, 1-penten-3-one, propanal, 2-propenal, 2-butenal, 3,5-octadiene, 2-pentenal, 2-ethylfuran, 2,4-heptadienal, 2,6-nonadienal. And again, oils from different raw materials, and natural oils versus concentrate oils, showed differences in their volatile compound profiles, reflecting differences in fatty acid composition.

### 3.6.3 TRIAL 3 – STORAGE EXPERIMENT

Eight refined oils selected from Trial 2 were analysed after 7, 14 and 20 days of storage. Again, the secondary lipid oxidation products found in Trial 1 and 2 dominated the volatile compound profiles of the oils. Compared to the fresh oil, the oils showed increasing levels of volatile secondary lipid oxidation products with increasing storage time, as shown for 2-ethyl furan and 1-penten-3-ol for two natural cod liver oils and two anchoveta concentrate oils (Figure 10).

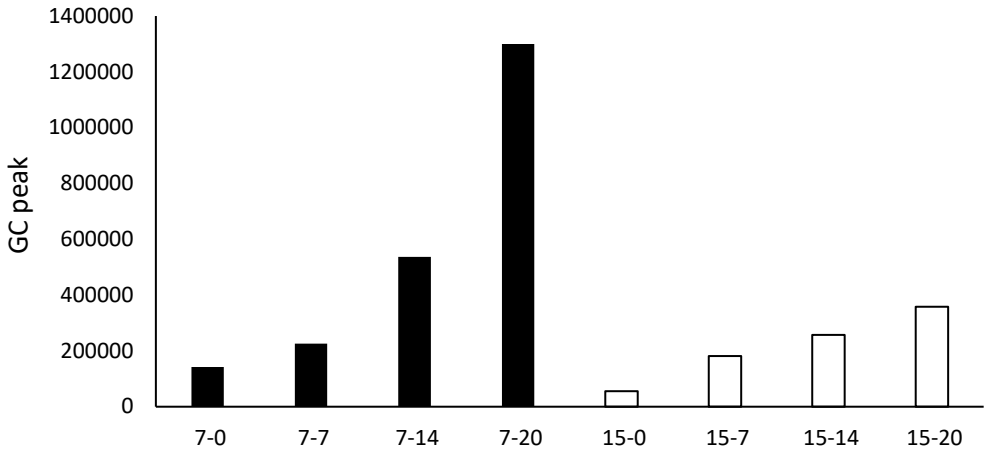


**Figure 10. 2-ethyl furan levels (GC peak area) in two cod liver oils (7 and 15) in samples stored for 0, 7, 14 and 20 days.**

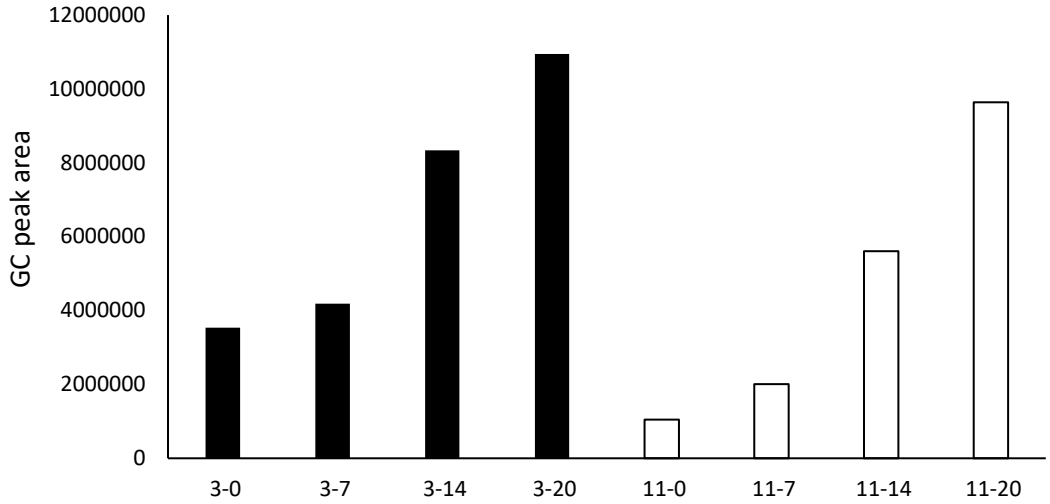


**Figure 11. 2-ethyl furan levels (GC peak area) in two anchoveta oil (3 and 11) in samples stored for 0, 7, 14 and 20 days.**

## 3.7 ASSOCIATION BETWEEN SENSORY PROPERTIES AND VOLATILE COMPOUNDS

### 3.7.1 TRIAL 1

*Screening of odour*

Trial 1 started with a sensory odour screening of all oils. The samples consisted of 28 oils in total, 18 refined and 10 undeodorized. Anisidine value (AnV) reflects mainly the amount of 2-alkenals and 2,4-dienals. There was no significant correlation (PLSR) between the volatile organic compounds and anisidine value (AnV) among any of the oils, not even within oil types (natural oils, concentrates, CLO and Anchoveta) or treatment. This may be explained by the fact that the different oil types have different fatty acid profiles, which influence the profile of volatile compounds since various volatile secondary oxidation products will be formed from different polyunsaturated fatty acids. In addition, there may be an effect of processing on the final volatile composition.

Volatile compounds correlated in general with high rancid sensory scores of the oils. Individual volatile secondary lipid oxidation products showed significant non-linear univariate correlations with rancid odour (r=0.62–0.82), and the highest correlation was found for 3,5-octadiene (Figure 12). 3,5-octadiene has a fruity, green, grassy smell.

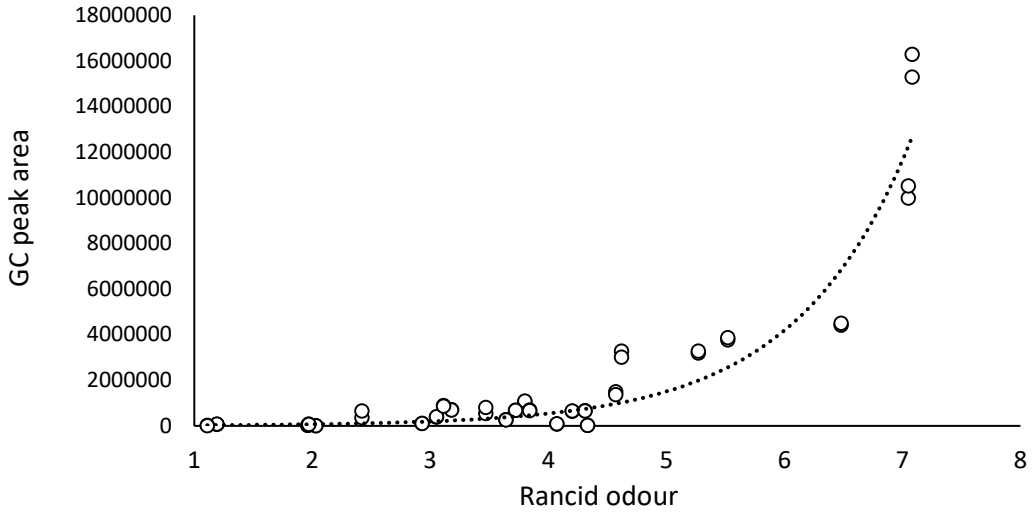


**Figure 12. Replicate GC peak areas of 3,5-octadiene versus rancid odour of all oil samples (r=0.82, p<0.0001).**

Multivariate regression (PLSR) between volatile compounds and sensory odour characteristics for all oils together showed significant correlations as follows: *process* r=0.90, *fish* r=0.89, *rancid* r=0.88 and *sourness* r=0.80 (0.0001).

*Profiling of odour, flavour, taste and mouthfeel*

After the screening, 16 oils – 12 refined and 4 undeodorized – went through an additional sensory profiling. The volatiles were dominated by secondary lipid oxidation products. Oils with high levels of volatile compounds also had high sensory scores for the *rancid* attribute.

Multivariate regression (PLSR) between volatile compounds and sensory odour characteristics for all oils together showed no significant correlations. PLSR of the refined oils only, however, gave significant correlations between the volatile compounds and sensory odour characteristics: *sourness* r=0.83, *fermented* r=0.81, *medicine* r=0.82, *rancid* r=0.80, *process* r=0.76 and *chemical* r=0.76 ($p<0.0005$) and flavour/taste characteristics: *bitter* r=0.87, *rancid* r=0.80, *process* r=0.80, *fermented* r=0.75 and *chemical* r=0.75, ($p<0.0005$).

### 3.7.2 TRIAL 2

Oils with high levels of volatile compounds correlated in general with high rancid sensory scores. However, no significant correlation (PLSR) between the volatile organic compounds and anisidine value (AnV) among any of the oils, not even within oil types (natural oils, concentrates, CLO and Anchoveta) or treatment, could be found for these data either.

Multivariate regression (PLSR) between the volatile compounds and sensory scores of all oils together gave a significant ($p<0.0005$) correlation for rancid and process odour and flavour (r=0.7–0.8). Anchoveta oils showed lower correlations between volatile compounds and sensory results, and the highest correlations were found for rancid and process odour and flavour (r=0.6, $p<0.0025$). The cod liver oils' volatiles had a high correlation ($r>0.9$, $p<0.0005$).

Correlation computations (PLSR) between sensory data and volatile compounds showed that the secondary lipid oxidation products 1-penten-3-ol, 2-ethylfuran, 1-penten-3-one, tr,2-pentenal, 2-propenal and propanal had the highest correlations (r=0.7–0.8, $p<0.0005$) with the sensory data.

### 3.7.3 TRIAL 3 – STORAGE EXPERIMENT

There was a significant correlation (PLSR) between the volatile organic compounds and anisidine value for all oil samples together (r=0.89, $p<0.0005$) and within oil types: r=0.89 ($p<0.0005$) for anchoveta oils, and r=0.99 ($p<0.0001$) for cod liver oils. The regression model for anisidine value

of the cod liver oils is shown in Figure 13.

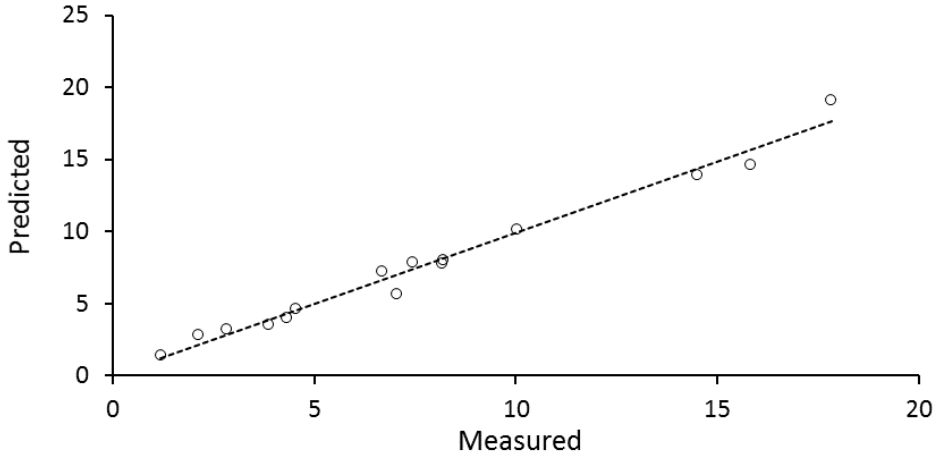


**Figure 13. VOC regression model for anisidine value of cod liver oil samples (r=0.99, p<0.0001).**

In general, oils from the storage experiment with high levels of volatile compounds correlated with high rancid sensory scores. The volatile compounds were dominated by the afore-mentioned typical secondary lipid oxidation products, indicating that lipid oxidation explains the major variance in the measurement data. This is in agreement with the sensory results, and was as expected given the storage conditions applied, i.e. room temperature and exposure to air.

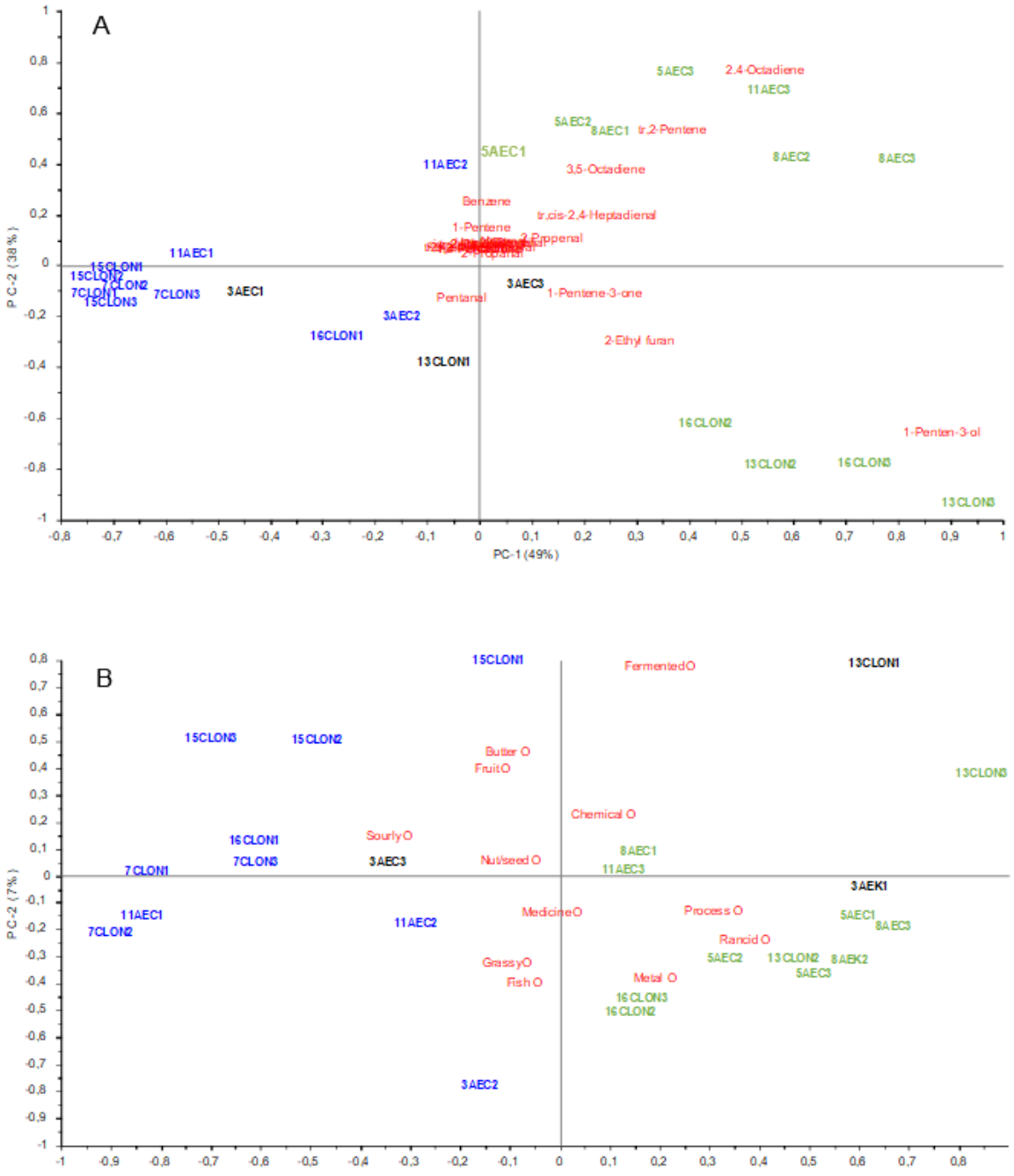


**Figure 14. Distribution (PCA biplot) of storage samples based on the volatile compounds (A) and the 12 odour attributes (B). For comparison, the blue and green colours show the similarity between the two figures.**

The variation in the composition of the sensory scores for the odour characteristics and volatile compounds of the samples from the storage experiment is shown in the distribution (PCA biplot) plots in Figure 14 for comparison. There is a good agreement in the sample distribution between the sensory and the chemical (volatile compounds) data; samples with low sensory rancid scores

and low levels of volatile secondary lipid oxidation products (in blue) are grouped along the left side, and samples with high sensory process and rancid scores and high levels of oxidation products (green) are grouped along the right side of the first component (PC-1), except for three samples (black): 3AEC1, 3AEC3 and 13CLON1.

The sum of volatile compounds showed a significant correlation with rancid odour ($r=0.80$, $p<0.0002$). Volatile compounds of all the oil samples together had a significant correlation with rancid odour ($r=0.80$, $p<0.0002$) and rancid flavour ($r=0.75$, $p<0.0002$). Anchoveta samples showed no correlation between the volatile compounds and sensory characteristics, but the cod liver oils showed a significant and high correlation with rancid ($r=0.91$, $p<0002$), process ($r=0.88$, $p<0.002$) and metal odour ($r=0.87$, $p<0.0002$), and for rancid flavour ($r=0.91$, $p<0.0002$) and process flavor ($r=0.88$, $p<0.0002$).

## 3.8 MARKET REQUIREMENTS

### 3.8.1 INTERVIEW OF PRODUCERS OF MARINE OILS

*Production and market segments*

The producers of marine oils offer several different products (Fig. 15). Nine of them produce pure oil in bulk. Six of the producers also add aroma, four produce bottled products of pure oil, one produces emulsions, one capsules and one soft chew.

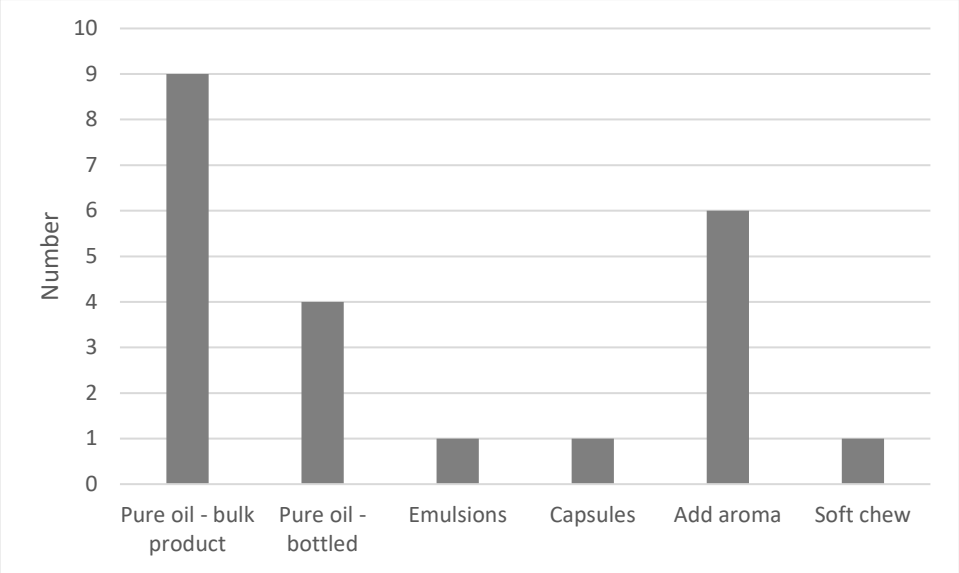


**Figure 15. Production of marine oil products among the companies.**

The products are sold to different market segments (Fig. 16). Nine of the producers sell to the health food/supplement segment, three to pharmacies, three to functional food producers, one to animal feed manufactures and one to cosmetics manufactures.

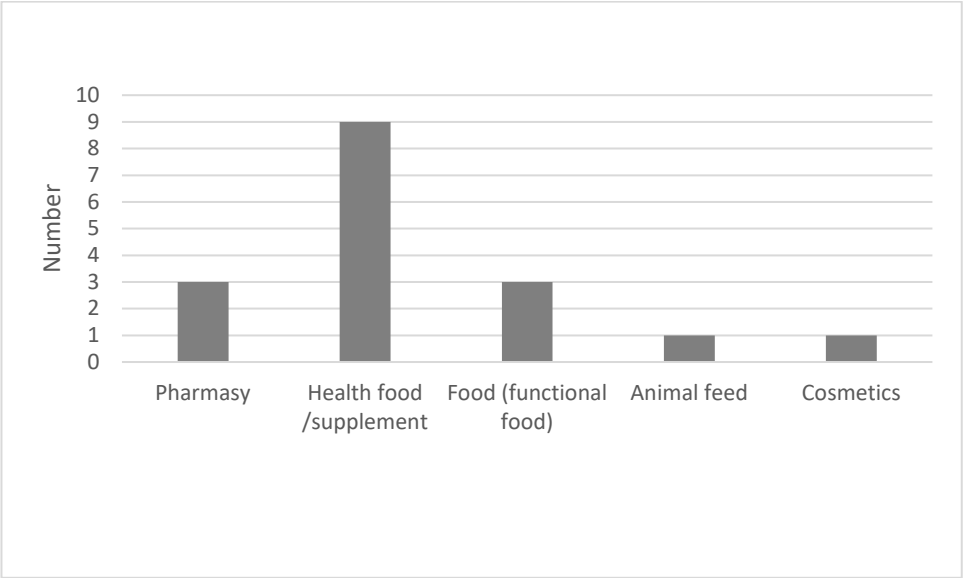


**Figure 16. Market segments.**

*Use of sensory methods internally in the company*

*Variation*

The survey showed quite large variations between the companies with regard to how they used sensory methods. In general, companies would use sensory methods during process control and in finished product testing. Approval of products, stability testing and anti-oxidant testing were also mentioned as areas of application. The variation correlated with whether the producer manufactured natural oils or concentrated oils, and whether they were primarily engaged in bulk oil manufacturing or also produced finished bottled products. There were also great variations in how detailed the methods used were and in the level of knowledge/training of the personnel responsible for the testing. Some companies also used sensory methods on finished products before adding flavour or antioxidants to the product. In most cases, some sensory testing was done in addition to the required chemical analysis.

*Production processes*

Bleaching, distillation, deodorization and filling were mentioned as process steps were sensory methods were used. Sensory evaluation was also used if there had been changes in the processing steps that should be controlled. A respondent claimed that increased use of nitrogen in the various processing steps, and better quality of the raw material used as starting material, had improved the quality of finished products.

*Level of knowledge and internal training*

Several of the companies reported a lack of specifically trained sensory panels and formalized routines for sensory methods and testing. Some companies reported that the need for sensory evaluation depended on the products being produced. In addition, some companies also

reported that the ability to perform sensory evaluations depended on what personnel was present at the time of testing. Some companies engaged in significant amounts of experience-based tasting, and who did the sensory tasting varied between e.g. technicians, R&D and production personnel. Long experience in producing marine oils had led to the establishment of specific terms and vocabulary for describing sensory characteristics, and established practice resulted in simplified sensory wheels and fewer categories for product testing. Producers running shifts also experienced challenges with regard to establishing and training qualified personnel, insofar as specific personnel would not be regularly available to perform testing.

On the opposite side, the survey revealed that several companies employed competent sensory panels with well-established routines for testing, advanced evaluation schemes, score systems, screening tests against reference samples, annual training sessions and sensory testing established as a strategic area of differentiation. These companies had clearly defined evaluation forms and were using established sensory wheels with minor adaptions to their own experience with regard to sensory evaluation. In new product-development processes, these companies used sensory characteristics and methods as part of the innovation process. Training in these companies was highly professional. For example, in one company “newcomers” to the panel would go through 10–12 specific tests to become enrolled.

There seems to be a relationship between level of knowledge, established routines, complexity of the products, end use of the oil, and market requirements when it comes to sensory practices.

In general, the companies were positive to developing a sensory standard, but the standard must be simple and easy because the personnel performing the tests have different skill levels and experience. Several of the companies wanted to increase their internal expertise, and commonly defined terms and standardizations among the companies are important.

### *Use of sensory characteristics and the sensory wheel*

The companies were asked what kind of sensory characteristics they used to describe the oils they produce, and to what extent they use the sensory wheel. As we can see in Figure 17, taste/odour is the most used characteristic; ten of the companies use this characteristic to describe oils. Smell and appearance are also important, while mouthfeel is less used.

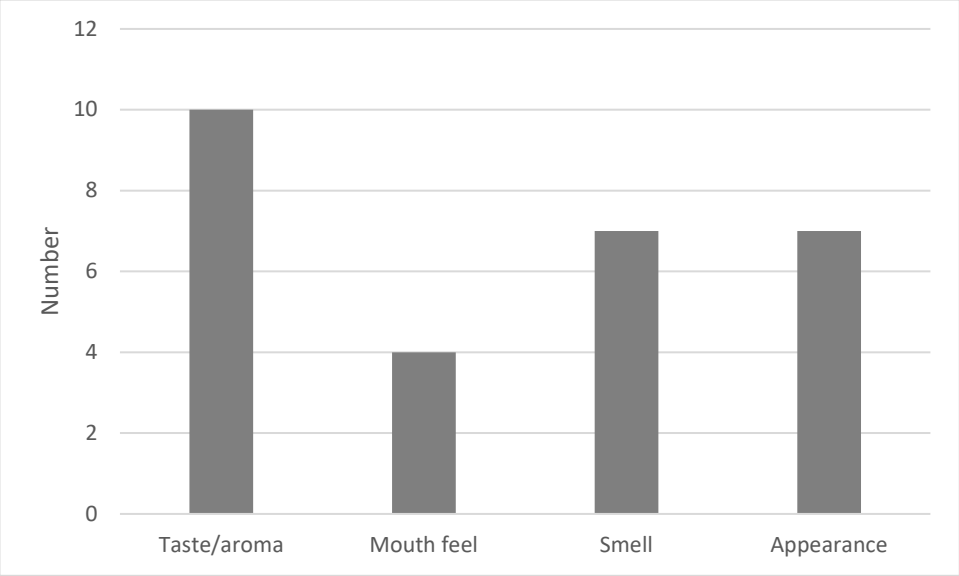


**Figure 17. Use of sensory characteristics in the companies.**

When it comes to use of the sensory wheel as a tool for describing the oils, companies used different amounts of nomenclature in their descriptions (Fig. 18). *Nuts* and *seeds*, *butter* and *grassy* were most frequently used to describe positive characteristics, and *fish*, *process* and *rancid* were most frequently used to describe negative characteristics.

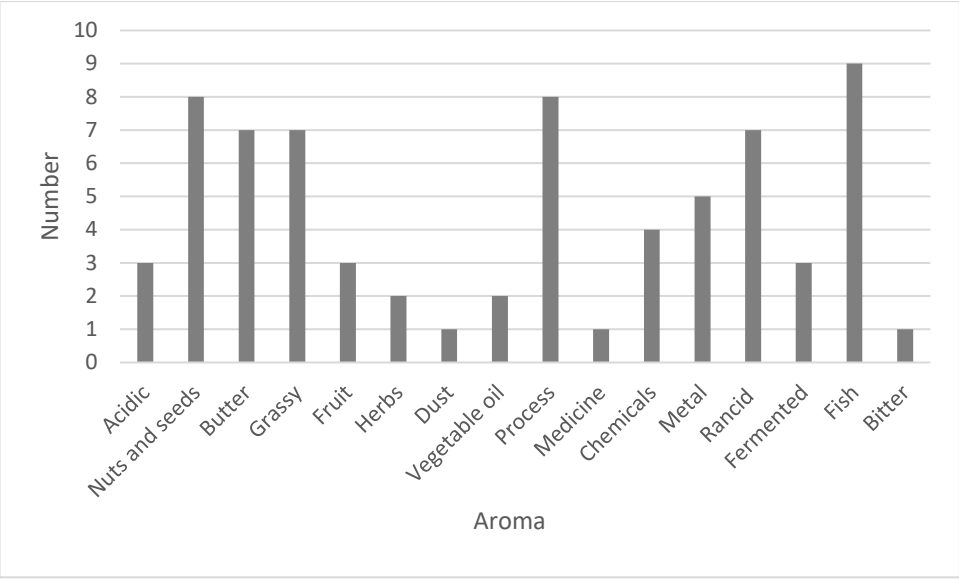


**Figure 18. Use of sensory nomenclature in the companies.**

*Customer requirements regarding sensory characteristics*

The customers of marine oil producers are different when it comes to requirements for sensory characteristics. There are few sensory requirements related to oil for capsule production. The producers say that there are more demands and questions regarding oil used in food, bottle products and chewable tablets. One of the respondents said that saturated and mature markets might lead to more demands regarding quality and more questions regarding sensory quality. Customers want product samples to assess quality, and using samples is more common than describing or documenting sensory characteristics. Customers lack concepts and training to assess taste, and it is therefore difficult for them to determine product quality based on sensory nomenclature. When customers are describing quality requirements regarding taste, they use different terms, and generally a simple vocabulary:

- degree of fish flavour or rancidity
- good, little taste (tasteless)
- mild
- fishy, but not rancid
- neutral taste
- as little taste and smell as possible
- taste- and odourless

*Benefits of having sensory quality standard*

The respondents found sensory quality standards to be beneficial in different ways, both internally and externally.

Internal benefits mentioned included having a more structural approach to testing that would reduce subjectivity and systematize the 'non-described' knowledge. A prerequisite for achieving this perceived benefit was that training in use of the standard was taken seriously and that all relevant personnel participated in regular training to build internal competence. An internal standard could have more categories than an external standard, but it was pointed out that it was necessary to keep it simple and use descriptions that were easily understood by operators and employees with different background knowledge. A standard would also help systematize internal work. New product-development processes could be simplified if a well-known standard was used.

Externally, it was pointed out that a standard could provide a common language to meet requirements from customers and thus simplify sales work by supporting market and sales communication. For some of the companies the information flow and communication between lab and sales personnel had been inadequate.

A standard could also be a tool to increase customer knowledge with regard to what to expect from a marine oil. Some respondents said that this could result in tougher requirements from customers, as well as, in some cases, push cost levels upwards due to 'unnecessary' processing steps to meet the requirements. A standard introduced to customers should in any case be simple.

It was commonly accepted among producers that sensory training reduced subjectivity. Hence, even though it was hard to train customers, sensory testing and use of sensory methods were considered to be an area of expertise that was important when building credibility and reputation. It would be a significant point of differentiation compared to competitors that are not using the standard, and a tool to build competitive advantages over foreign competitors.

*Other topics*

Other important issues mentioned by the companies concerned how customers handled the marine oils after delivery (b2b), and how this may affect the oil quality and the end consumer perception of the taste. Correct storage of the oils is important to maintain the quality of the oil. For example, too high storage temperatures might affect the oil negatively. Another question was how different anti-oxidants might affect the taste during storage. Use of aroma was also important: What aromas can be used on fish oil and what impact might they have on different types of fish oil?

Among other topics mentioned was concerns regarding the stability of the oil – what the oil can tolerate and what kind of antioxidants suited each production process. Some respondents also mentioned that they wanted more knowledge about the different components in the oil that may affect taste. Packaging and storage factors must also be included in the assessment of sensory qualities.

### 3.8.2 SURVEY AND INTERVIEW WITH CUSTOMERS OF PRODUCERS OF MARINE OILS

The customers of the oil producers vary significantly when it comes to how they use the oil. Some manufacture products based on the fish oil, whereas others are just distributors to other production companies. Some of them have insufficient knowledge of sensory characteristics like taste. The distributors might conduct small tests, such as evaluating colour (appearance) and smell. Customers who produce end products perform more thorough and professional sensory analyses.

*Production and market segments*

Different segments have different requirements and needs. One company said that in products aimed at children the oil should have less smell and thermal stability guaranteed. For adults and elderly customers, appearance should be transparent and clear. For functional food the contents of EPA, DHA are important. One company said that children prefer chewing capsules, and that this is also an alternative for elderly people.

*Customer requirements regarding sensory characteristics*

One of the companies said that they get questions from their customers in particular regarding taste, smell and appearance. They also said that there were different requirements for sensory characteristics/attributes based on type of customer and even on type of product (pure oil or capsule).

*Use of sensory methods internally in the company*

Whether the customers use sensory testing varies. Some of the companies produce products based on the fish oil, and for these it is very important to do sensory testing.

One of the companies who produce dietary supplements have an in-house sensory panel. Tests take place in a special room and take place regularly (biweekly) with 5 to 8 trained persons. They test flavour, mouthfeel, odour, appearance and viscosity. When it comes to sensory characteristics, they use the following vocabulary: Herbs, chemicals, metal, rancid, fermented, fish, bitter and sulphurous. For mouthfeel they use: Hot, astringent, thin, thick, fatty, clumpy.

One of the distributors say they are also conscious regarding how to store marine oils. They store the oil in the original packaging under nitrogen, and they have procedures in place to ensure containers are not opened by customs for inspection. Warehouse is temperature controlled and kept below 30 °C.

*Importance of a sensory quality standard*

One of the companies said that a quality standard will provide an objective way to determine the sensory qualities of the products they sell. Sensory qualities are also important for marketing and innovation themes.

*Other topics*

Regarding other topics, companies mentioned documentation of heavy metals, dioxin etc., based on the manufacturer's CoA and contents of EPA/DHA. One company also said that country of origin was important.

## 3.9 CLASSIFICATION AND STANDARDIZATION

The differences between the classes are based on a total of eight sensory characteristics (Table 13). Four characteristics are rated as being accepted deviations from an odour- and flavour/taste-less fish oil, while four characteristics are only accepted in very low (1) or moderate (2) intensities. In addition, the sensory panels can name other characteristics, but the presence of these must also be of a low or moderate intensity.

**Table 13. Acceptable, and less acceptable sensory characteristics used in the classification**

| **Acceptable** | **Acceptable at a very low to low intensity** |
|---|---|
| Sourness | Fish |
| Grass | Fermented |
| Butter | Rancid |
| Nut | Process |
| | Other (i.e. chemical, metal or pungent) |

The intensity of the characteristics is suggested measured by a 5-point intensity scale from 0 to 4, where 4 is high intensity (see Table 4).

The sensory evaluation is done by using an adjusted quality control test (NMKL:201 2017), and score and classification are connected as illustrated in Table 14. It is suggested that a product specification is used instead of a reference oil. The product specification refers to an odour- and flavour/taste-less oil with no specific sensory characteristics. Minimal deviation (4) from the product specification equals GOLD, extra high sensory quality, and may include accepted sensory characteristics such as *sourness*, *grass*, *nut* and *butter*. A low intensity of fish odour and flavour/taste is also allowed. Weak deviations (3) from the product specification equals SILVER, high sensory quality. A SILVER oil may in addition to the accepted characteristics have a low intensity of fish, fermented, process and rancid odour and flavour. Moderate deviations from the product specification equals REGULAR sensory quality, and may in addition to the accepted characteristics have a moderate intensity of fish, fermented, process and rancid odour and flavour/taste.

**Table 14. Suggestion of alteration of the score scale of assessment of fish oils in quality control tests NMKL 201 (NMKL:201 2017).**

| Point | Deviation to product specification | Accepted deviation | Classification |
|---|---|---|---|
| 5 | No deviation. | Odour- and flavourless | **GOLD** (Extra high sensory quality) |
| 4 | Minimal deviation from product specification | Sourness, grass, nut and butter (present)<br>Fish (low intensity) | |
| 3 | Weak deviation from product specification | Sourness, grass, nut and butter (present)<br>Fish, rancid, fermented and process (low intensity)<br>Other (e.q. chemical, metal, fruit) (low intensity) | **SILVER** (High sensory quality) |
| 2 | Moderate deviation from product specification | Sourness, grass, nut and butter (present)<br>Fish, rancid, fermented and process (moderate intensity)<br>Other (e.q. chemical, metal, fruit) (moderate intensity) | **REGULAR** sensory quality |
| 1 | Distinct deviation from product specification | Deviation not included in the standard | Not commodity |

A preliminary Norwegian common standard focusing on the sensory demands of these three classifications has been developed. The standard will be published in 2019–2020 and focuses primarily on the sensory qualities of the fish oils. These are summarized in Table 15. Peroxide values and anisidine values are included and based on GOED's recommendations. The allowed intensities of the sensory characteristics are shown in the four bottom rows.

**Table 15. Suggestion of classification of fish oils including chemical demands and accepted sensory characteristics (odour and flavour).**

| *Sensory characteristics and chemical parameters* | **GOLD** Extra high sensory quality | **SILVER** High sensory quality | **REGULAR** Sensory quality |
|---|---|---|---|
| Peroxide value | ≤5 | ≤5 | ≤5 |
| Anisidine value | ≤20 | ≤20 | ≤20 |
| Sourness, grassy, butter, nut | Allowed | Allowed | Allowed |
| Fish (fresh) | ≤1 | ≤1 | ≤2 |
| Fermented, rancid, process | 0 | ≤1 | ≤2 |
| Other (i.e. chemical, metal, pungent, fruit) | 0 | ≤1 | ≤2 |

The classification of the oils in trail 1 and 2 is showen in appendic 1.

## 3.10 SIGNIFICANT DIFFERENCES BETWEEN CLASSIFICATION AND SOME CHEMICAL PARAMETERS

As described earlier, a correlation between sensory and chemical analysis has been made. Applying this classification system to the oils delivered in the project, we see that all the newly refined oils tested in the project are well below GOED's recommendations regarding anisidine- and peroxide values (se 3.3). But the results show that even if there is a correlation between higher oxidation values and rancid flavour, and between lower oxidation values and the characteristic sourness, it has not been possible to suggest recommendation levels for primary (pV) and secondary (AnV) oxidation connected to the different classes. Table 16 shows the average and the max/min. values of peroxide, anisidine, free fatty acids, colour, total volatiles and some selected volatiles connected to the three classifications. There is a wide overlap between the classifications and the max/min. values. Products with GOLD (class A) extra high sensory quality have on average a lower peroxide value and total volatiles compared to products in the other two classifications, but only the peroxide value exhibits a weakly significant difference between classes GOLD and SILVER ($p<0.01$) and between GOLD and REGULAR ($p<0.01$). There is no significant difference between classifications for FFA, AnV or colour.

The GOLD (class A) products have a significantly lower concentration of the volatiles 1-penten-3-one, 2-pentenal and 2-ethylfuran than in products in classification C (REGULAR) ($p<0.01$).

**Table 16. Average and min/max values of peroxide, anisidine, free fatty acids, colour, total volatiles and the average of selected volatiles 1-penten-3-ol, 1-penten-3-one, propanal, 2-propenal, 2-butenal, 3,5-octadiene, 2-pentenal, 2-ethylfuran and 2,4-heptadienal connected to the three classifications.**

| | **Classification** | **GOLD** | **SILVER** | **REGULAR** |
|---|---|---|---|---|
| | PV (meq peroxide/kg oil) | 0.6 (0.2–1.4)[a] | 2.0 (0.3–4.0)[b] | 2.0 (0.4–4.1)[b] |
| | AnV | 5.3(1.9–13.9) | 5.4 (2.8–14.0) | 6.3 (1.4–14.9) |
| | FFA (%) | 0.1 (0–0.2) | 0.1(0–0.3) | 0.2 (0–0.7) |
| | Colour | 3.0 (1.5–4.3) | 3.0 (1.0–4.3) | 2.6 (1.0–5.0) |
| GC peak areas | Total volatiles (E+07) | 3.37 (0.24–6.57) [a] | 12.08 (0.31–58.25) [b] | 15.93 (1.28–58.86) [b] |
| | 1-penten-3-ol | 5.40E+06 | 7.70E+06 | 2.50E+07 |
| | 1-penten-3-one | 1.50E+06[a] | 3.20E+06[ab] | 8.70E+06[b] |
| | Propanal | 9.00E+05 | 1.40E+06 | 2.70E+06 |
| | 2-propenal | 4.40E+06 | 4.80E+06 | 1.10E+07 |
| | 2-butenal | 3.00E+05 | 5.00E+05 | 3.60E+06 |
| | 3,5-octadiene | 1.80E+06 | 2.50E+06 | 8.30E+06 |
| | 2-pentenal | 0.07+06[a] | 1.20E+06[ab] | 6.40E+06[b] |
| | 2-ethylfuran | 1.50E+06[a] | 2.80E+06[ab] | 9.40E+06[b] |
| | 2,4-heptadienal | 1.40E+05 | 1.60E+05 | 1.20E+06 |

Values with a different letter (a-b) within a row for the same chemical parameter are significantly different ($p<0.01$)

# 4. DISCUSSION

The aim of this study has been to identify the most important descriptors of fish oil and connect these to a common sensory standard. When identifying sensory descriptors it is important to have a selection of samples that covers different sources of variation and encompasses a wide range of sensory attributes (Drake et al. 2002). The 70 different fish oils produced by eight different companies represented a broad selection of the available fish oil products on the market, and were based on a selection of raw materials caught in both Norwegian and foreign waters (Table 1). The same approach was used in Koch et al. (2012), discussing 69 different samples of rooibos tea from 64 producers, and Theron et al. (2014), which included 58 samples from six different honey bush species when identifying sensory characteristics.

To ensure a wide sensory variation in the present study, the producers delivered products with specific fatty acid composition and oils used both in functional food, as drinking oil and for capsule production. Ten of our oils had not gone through deodorization. These would normally not have reached the consumer. The sensory characteristics of the oils correspond well with earlier investigations of marine oils (Larssen et al. 2018).

The wide range of samples and qualities resulted in a suggestion of three sensory classifications of fish oils. In a further study it may be interesting to characterize the oils in the different classes separately to get a better separation and understanding of the different classes. This strategy was used by Aparicio et al. (1996). When investigating the relationship between volatile components and sensory attributes in 16 olive oil samples, Aparicio et al. chose to include only virgin olive oil, and exclude extra virgin and pomance oils. Even if the variety of fish oils in the present study was broad, other oils produced from other raw materials or using other processing methods may have other sensory characteristics. The sensory and chemical attributes that are described in this study are nevertheless evaluated as providing a broad enough selection for the standardization work.

The sensory profiling of the fish oils was conducted by nine professional assessors. In addition, all the nine industry partners participating in the study and some of their customers were interviewed regarding which types of sensory attributes were most common, most important for sensory quality and most important for meeting requirements from the market. Aparicio et al. (1996) chose to use six different professional panels consisting of five different nationalities and different types of experience (Communities 1991, ISO 1993) when testing olive oil, while Hersleth et al. (2005) used five expert assessors to evaluate cheese before it was profiled by a trained sensory panel. Gawel et. al (2000), characterizing mouthfeel in red wine, and Theron (2012), developing sensory profiling for Cyclopia Species (Honeybush), chose to use a trained sensory panel for the language development sessions. Neither used expert panels.

To specify the most important attributes of the fish oils, the sensory wheel published by Larssen et al. (2018) was used as a starting point. The sensory wheel consists of 21 main categories and 60 keywords. In the sensory profiling 22 different characteristics (10 odours and 12 flavours/tastes) were used. This is in line with the number of attributes recommended by

Vannier et al. (1999) for the purpose of efficient sensory profiling. Through the interviews with the industry together with the sensory profiling done by a trained sensory panel, four sensory characteristics were defined as *accepted* and allowed in the fish oils regardless of intensity and classification. In addition, four main deviations and the category 'others' are defined as being allowed at a specific level intensity in the different classes. As this study is one of the first seen in the literature that discusses and organizes sensory attributes of fish oils it is important to capture as much as possible of the sensory variations in and among the different oils; modification and grouping of the attributes were accordingly necessary.

Even though the reduction of sensory characteristics in the sensory standard is large, the work done by Larssen et al. (2018) is important, and it is recommended as a basic tool for analysing the sensory qualities of a fish oil. This is in agreement with the experiences of other authors stating that a rigid reduction of descriptors could result in a loss of specific attributes that would be essential in characterizing the unique sensory profiles of the product (Wolters and Allchurch 1994, Theron et al. 2014).

In a future study a correlation between the selected characteristics and the suggested classifications should be investigated in the same way as was done in studies of sensory profiling of olive oil (Mojet and de Jong 1994, Monteleone and Langstaff 2014).

The marine oil industry usually separates their sensory characteristics into positive and negative attributes during quality control. The PCA plot (Figure 2) gives the location of sourness, butter, nut and grass aroma and tastes on the left side of the plot, while metal, rancid, fish and process aromas and tastes are located on the right side of the plot. The correlation between the 'positive' attribute *sourness* and the 'negative' attribute *rancid* confirms the industry's experiences. This is in accordance with Larssen et al. (2018). The negative attributes are usually evidence of unsuccessful refining, raw materials of low quality or inadequate storage. For olive oil, wine and beer defects wheels including negative attributes have been developed (Langstaff 2009, Langstaff 2009, Langstaff et al. 2011). These wheels can be useful for detecting errors during production or storage. A similar wheel could also be beneficial for the marine oil industry.

A PCA loadings plot can also be used to investigate whether some attributes used in the profiling are redundant, thus reducing or simplifying the set of terms, and also preventing different attributes from being used to describe identical sensory characteristics (Næs et al. 2010). The PCA loadings plot can also demonstrate whether correlations exist between aroma and flavour attributes that have been analysed by nose (orthonasal, ON) and by mouth (retronasal, RN), respectively. Most of the ON and RN attributes in this study (like *sourness*, *fish*, *rancid*, *process*) were closely associated with each other, which indicates that these notes were perceived similarly in the nose and on the palate. Accordingly, in a further study it may be possible to use odour as a first scan and to roughly sort the samples. Development of synthetic odour references may also be of great help for the industry in their work regarding sensory classification and standardization.

No preference testing of oils was conducted in the study. In a further study preference testing after classification by the new system may be beneficial to evaluate whether the sensory classification is consistent with market requirements.

Partial least squares regression (PLSR) shows that the sensory characteristics *rancid, chemical, metal* and *process* are positively correlated with high peroxide and anisidine values. Earlier studies have shown that fresh marine oils correlate with the sensory characteristics *fish, sweet, grass* and *butter*, while stored marine oils with increased peroxide and anisidine values correlate with *acidic, metallic, pungent* and *paint* (Serfert et al. 2010).These results correspond to the findings in our study, except for *sourness* (*acidic)* flavour. *Acidic* flavour has earlier been described as a sensory attribute of fresh sunflower oil (Serfert et al. 2010).

The fish oil samples studied contained up to about 100 volatile compounds, of which about 80% could be identified. They were dominated by volatile secondary lipid oxidation products from unsaturated fatty acids: 1-penten-3-ol, 2,4-octadiene, 1-penten-3-one, tr,2-pentenal, 3,5-octadiene, 2-pentene, propanal, tr,2-propenal, tr,2-butenal, 2-ethyl furan, tr,cis,2,4-heptadienal, hexanal and acetic acid. Oils with the highest intensity of rancid odour and taste also had the highest levels of these secondary lipid oxidation products. Significant correlations were found between volatile compounds and the characteristics *rancid*, *process*, *sourly*, *fermented*, *medicine* and *metal*, and the flavour characteristics *process*, *fermented*, *bitter*, and *chemical*. Poor correlations were in general found between the volatiles and anisidine value when oils of various types (anchoveta, cod liver oil, natural, concentrates) were combined; within each type of oil, highly significant correlations were for the most part obtained. This is explained by the variation in fatty acid profiles depending on oil raw material and processing, i.e. whether the oils are natural or concentrate, since different fatty acids profiles generate different volatile secondary lipid oxidation products. In addition, the content of anti- and pro-oxidants as α-tocopherol, retinol, carotenoids, polyamines, phospholipids, peptides and trace metals in crude oils will also vary with species and which will affect the composition of volatile compounds in refined oils. However, no systematic data exist on the comparison of volatile compound profiles and levels in fish oils from different fish species raw material, except for a study made by Giogios et al. (2009). They demonstrated also different volatile profiles depending on the raw material and fish species in secondary lipid oxidation products in relation to fatty acid composition.

Based on the data collected in this study it is possible to differentiate between the classes for some of the chemical oxidation products. The GOLD class products have significantly lower values of peroxide and total volatiles compared to products in the classes SILVER and REGULAR. GOLD class products also have significantly lower concentrations of 1-penten-3-one, 2-pentenal and 2-ethylfuran compared to REGULAR products. Despite this, no stricter chemical demands connected to the classification and standardization have been suggested. This is the same practice used by the IOC (International Olive Council) in their sensory quality control system for olive oil (Monteleone and Langstaff 2014).

## 5. CONCLUSION

The study has shown that the sensory characteristics of fish oils give an accurate representative description of the quality of the oils and that a common sensory standard may be a valuable tool in the industries' quality control and marketing. A classification system of the fish oils is defined, providing the industry with a simple and convenient tool in communication with customers. Samples with low primary and secondary oxidation were associated with sensory attributes like *sourness* and *grass*, while oils with higher values along the oxidation parameters were associated with sensory attributes like *rancid*, *fermented* and *process*. The sensory characteristic *fish* is defined as the fresh odour and flavour of fish. This attribute is allowed in all classifications, but at a low intensity level. In a further study it may be beneficial to produce synthetic reference oils to train the sensory industries panels.

## REFERENSE


Aas, G. H., T. Barnung, M. Kjerstad and W. E. Larssen (2016). Fresh or frozen storage of Atlantic mackerel (Scomber scombrus)– effect on oxidation and sensory quality of canned mackerel fillet. I: . . Sustainable bio-resources- Management, product development and raw material quality. B. Thu and A. C. Gundersen, Orkana Forlag, Stamsund**:** (in pres).

AOCS (1997). Official Method Cd 8-53. Surplus 2003. Peroxide Value. Official methods and recommanded practices of the AOCS, American Oil Chemists Society (Champaign, IL, USA).

AOCS (2003). Flavour panel evaluation of vegetable oils. AOCS Recomended Practis Cg 2-83. Sampling and analysis of commercial fats and oils., American Oil Chemists' Society.

AOCS (2003). Official method Cd 18-90. p-Anisidine Value. Reaproved 1997. Official methods and recommended practices of the AOCS. American Oil Chemists Sosiety (Champaign, IL, USA).

AOCS (2009). "AOCS Official Method Td 1a-64. Color, Gardner 1963 (Glass Standards), Reapproved 2009

" Official methods and recommended practices of the AOCS. **American Oil Chemists Society**(Champaign, IL, USA).

AOCS (2009). Official Method Ce 1b-89 : Fatty Acid Composition of Marine Oils by GLC. Reapproved 2009, American Oil Chemists Sosiety (Champaign, IL, USA).

Aparicio, R., M. Morales and M. Alonso (1996). "Relationship between volatile compounds and sensory attributes of olive oils by the sensory wheel." Journal of the American Oil Chemists' Society **73**(10): 1253-1264.

Caporale, G., S. Policastro, A. Carlucci and E. Monteleone (2006). "Consumer expectations for sensory properties in virgin olive oils." Food Quality and Preference **17**(1): 116-125.

CODEX:19 (2009a). Codex standard for edible fats and oils not covered by individual standards. CODEX STAN 19-1981, Codex Alimentarius Commission**:** 1-5.

CODEX:33 (2009b). Codex standard for olive oils and olive pomance oils. Codex Stand 33-1981, Codex Alimentarius Commission**:** 8.

CODEX:210 (2011). Codex standard for named vegetable oils, CODEX STAN 210-1999, Codex Alimentarius Commission. **8:** 1-16.

CODEX:132303 (2013). Proposed draft standard for fish oils. Joint FAO/WHO food standards programme. Italia, CODEX Alimentarius Commission**:** 12.

Communities, E. (1991). Official Journal of the Commission of the Official Journal of the Commission of the European Communities. Regulation no 2668/91. Brussels, hentet i fra Aparicio *et al*. 1996**:** 48-74.

Drake, M., P. Gerard, S. Wright, K. Cadwallader and G. Civille (2002). "Cross validation of a sensory language for Cheddar cheese." Journal of sensory studies **17**(3): 215-227.

Gawel, R. and D. A. Rogers (2009). "The relationship between total phenol concentration and the perceived style of extra virgin olive oil." Grasas y aceites **60**(2): 134-138.

Giogios, I., K. Grigorakis, I. Nengas, S. Papasolomontos, N. Papaioannou and M. N. Alexis (2009). "Fatty acid composition and volatile compounds of selected marine oils and meals." Journal of the Science of Food and Agriculture **89**(1): 88-100.

Hersleth, M., M. A. Ilseng, M. Martens and T. Næs (2005). "Perception of cheese: a comparison of quality scoring, descriptive analysis and consumer responses." Journal of food quality **28**(4): 333-349.

ISO8586 (2012). Sensory analysis–general guidelines for the selection, training and monitoring of selected assessors and expert sensory assessors. International Organization for Standardization, International Organization for Standardization Geneva, Switzerland.

ISO (1993). Sensory analysis—General guidance for selection, training and monitoring of assessors, International Organization for Standardization Geneva, Switzerland.

Jacobsen, C. (1999). "Sensory impact of lipid oxidation in complex food systems." Lipid/Fett **101**(12): 484-492.

Koch, I. S., M. Muller, E. Joubert, M. van der Rijst and T. Næs (2012). "Sensory characterization of rooibos tea and the development of a rooibos sensory wheel and lexicon." Food Research International **46**(1): 217-228.

Langstaff, S. A. (2009). "The defects wheel for beer." Retrieved 02.08.16, from http://www.appliedsensory.com/defects-wheels.html.

Langstaff, S. A. (2009). "The defects wheel for wine." Retrieved 02.08.16, from http://www.appliedsensory.com/defects-wheels.html.

Langstaff, S. A., R. Aparicio and C.-S. Group (2011). "The defects wheel for olive oil." Retrieved 02.08.16, from http://www.appliedsensory.com/defects-wheels.html.

Larssen, W. E., E. Monteleone and M. Hersleth (2018). "Sensory description of marine oils through development of a sensory wheel and vocabulary." Food Research International **106**: 45-53.

Lawless, H. T. and H. Heymann (2010). Sensory evaluation of food: principles and practices, Springer.

Mojet, J. and S. de Jong (1994). "The sensory wheel of virgin olive oil." Grasas y aceites **45**(1-2): 42-47.

Monteleone, E., G. Caporale, A. Carlucci and M. Bertuccioli (1996). "Prediction of virgin olive oil sensory profile." Industrie Alimentari **35**: 1066-1072.

Monteleone, E., G. Caporale, L. Lencioni, F. Favati and M. Bertuccioli (1995). "Optimization of virgin olive oil quality in relation to fruit ripening and storage." Developments in Food Science **37**: 397-418.

Monteleone, E. and S. A. Langstaff (2014). Olive Oil Sensory Science, Wiley Blackwell.

Morales, M. T., M. V. Alonso, J. J. Rios and R. Aparicio (1995). "Virgin olive oil aroma: relationship between volatile compounds and sensory attributes by chemometrics." Journal of Agricultural and Food Chemistry **43**(11): 2925-2931.

NMKL:201 (2017). Quality control test- Sensory evaluation of marine oils. Nordisk metodikkomitè for næringsmidler., Nordisk metodikkomitè for næringsmidler.**:** 46.

Næs, T., P. B. Brockhoff and O. Tomic (2010). Statistics for Sensory and Consumer Science, Wiley Online Library.

Olsen, E. (2005). Analysis of early lipid oxidation in foods with n-3 fatty acids. Dept. of Chemistry, Biotechnology and Food Science, Norwegian University of life Sciences. **Doctor Scientarium Thesis 2005:26:** 73.

Ruyter, B., S. Grimmer, T. Thorkilsen, M. Todorcevic, M. Lalic and G. Vogt (2010). Light oxidized omega 3 oils and potential health benefits. Norwegian title: Lite oksiderte omega 3 oljer og potensielle helsefordeler, Nofima**:** 65.

Serfert, Y., S. Drusch and K. Schwarz (2010). "Sensory odour profiling and lipid oxidation status of fish oil and microencapsulated fish oil." Food chemistry **123**(4): 968-975.

Theron, K., M. Muller, M. Van Der Rijst, J. Cronje, M. Le Roux and E. Joubert (2014). "Sensory profiling of honeybush tea (Cyclopia species) and the development of a honeybush sensory wheel." Food Research International **66**: 12-22.

USDA (2010). United States Standards for Grades of Olive Oil and Olive-Pomace Oil, US. Department of Agricultur**:** 19.

USDA (2012). Grading Manual for Olive Oil and Olive-Pomace Oil, United States Department of Agricultur**:** 31.

Vannier, A., O. X. Brun and M. H. Feinberg (1999). "Application of sensory analysis to champagne wine characterisation and discrimination." Food Quality and Preference **10**(2): 101-107.

Wolters, C. J. and E. M. Allchurch (1994). "Effect of training procedure on the performance of descriptive panels." Food Quality and Preference **5**(3): 203-214.

## APPENDIC 1

| | Code | Collected | Main raw material | Composition | Classification |
|---|---|---|---|---|---|
| TRIAL 1 | 1CN_17 | 2017 | Cod liver | Natural | B |
| | 3AEC_17 | 2017 | Anchoveta | EPA concentrate | C |
| | 4AEC_17 | 2017 | Anchoveta | EPA concentrate | |
| | 5ADC_17 | 2017 | Anchoveta | DHA concentrate | A |
| | 6ADC_17 | 2017 | Anchoveta | DHA concentrate | B |
| | 7CN_17 | 2017 | Cod liver | Natural | |
| | 8CC_17 | 2017 | Cod liver | Concentrate | B |
| | 12ADC_17 | 2017 | Anchoveta | DHA concentrate | A |
| | *13ADC_17* | *2017* | *Anchoveta* | *DHA concentrate* | A |
| | 15CN_17 | 2017 | Cod liver | Natural | |
| | 16CN_17 | 2017 | Cod liver | Natural | B |
| | 17AEC_17 | 2017 | Anchoveta | EPA concentrate | A |
| | 18CN_17 | 2017 | Cod liver | Natural | |
| | 19CN_17 | 2017 | Cod liver | Natural | |
| | 20AN_17 | 2017 | Anchoveta | Natural | A |
| | 21AEC_17 | 2017 | Anchoveta | EPA concentrate | |
| | 22AEC_17 | 2017 | Anchoveta | EPA concentrate | B |
| | 23AEC_17 | 2017 | Anchoveta | EPA concentrate | B |
| TRIAL 2 | 1AN_18 | 2018 | Anchoveta | Natural | A |
| | 2AEC_18 | 2018 | Anchoveta | EPA concentrate | C |
| | 3AEC_18 | 2018 | Anchoveta | EPA concentrate | C |
| | 4TDC_18 | 2018 | Tuna | DHA concentrate | A |
| | 5AEC_18 | 2018 | Anchoveta | EPA concentrate | B |
| | 6ADC_18 | 2018 | Anchoveta | DHA concentrate | B |
| | 7CN_18 | 2018 | Cod liver | Natural | A |
| | 8AEC_18 | 2018 | Anchoveta | EPA concentrate | C |
| | 9AEC_18 | 2018 | Anchoveta | EPA concentrate | A |
| | 10ADC_18 | 2018 | Anchoveta | DHA concentrate | C |
| | 11AEC_18 | 2018 | Anchoveta | EPA concentrate | A |
| | 12CN_18 | 2018 | Cod liver | Natural | B |
| | 13CN_18 | 2018 | Cod liver | Natural | C |
| | 14AEC_18 | 2018 | Anchoveta | EPA concentrate | C |
| | 15CN_18 | 2018 | Cod liver | Natural | B |
| | 16CN_18 | 2018 | Cod liver | Natural | B |
| | 17AEC_18 | 2018 | Anchoveta | EPA concentrate | D |
| | 18ADC_18 | 2018 | Anchoveta | DHA concentrate | A |

MØREFORSKING AS
Postboks 5075
6021 Ålesund
TEL +47 70 11 16 00
www.moreforsk.no
NO 991 436 502